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of the
SOCIETY OF DYERS AND COLOURISTS

Volume 68 Number 11

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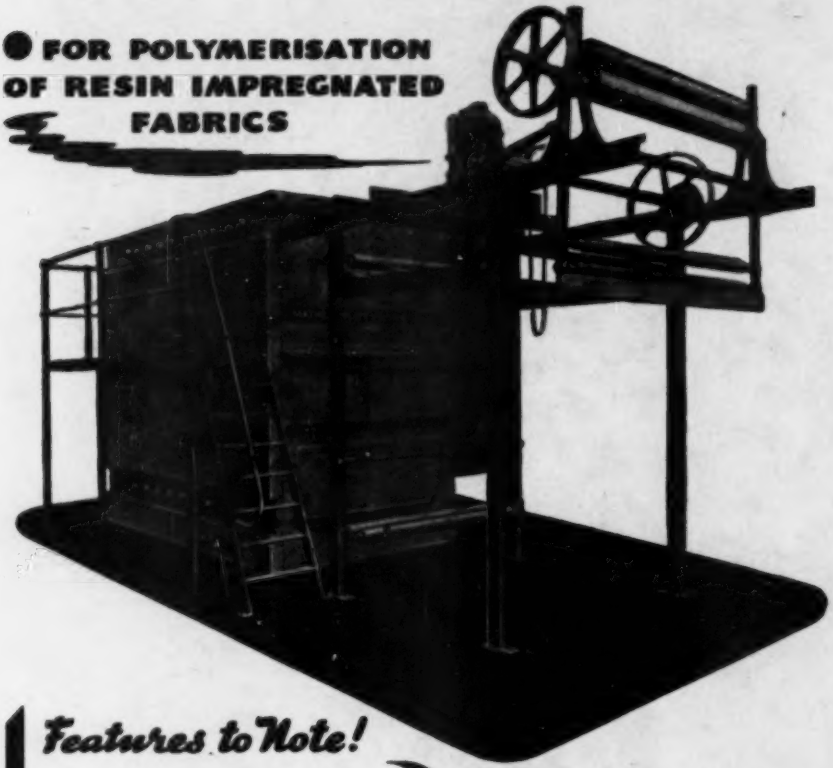

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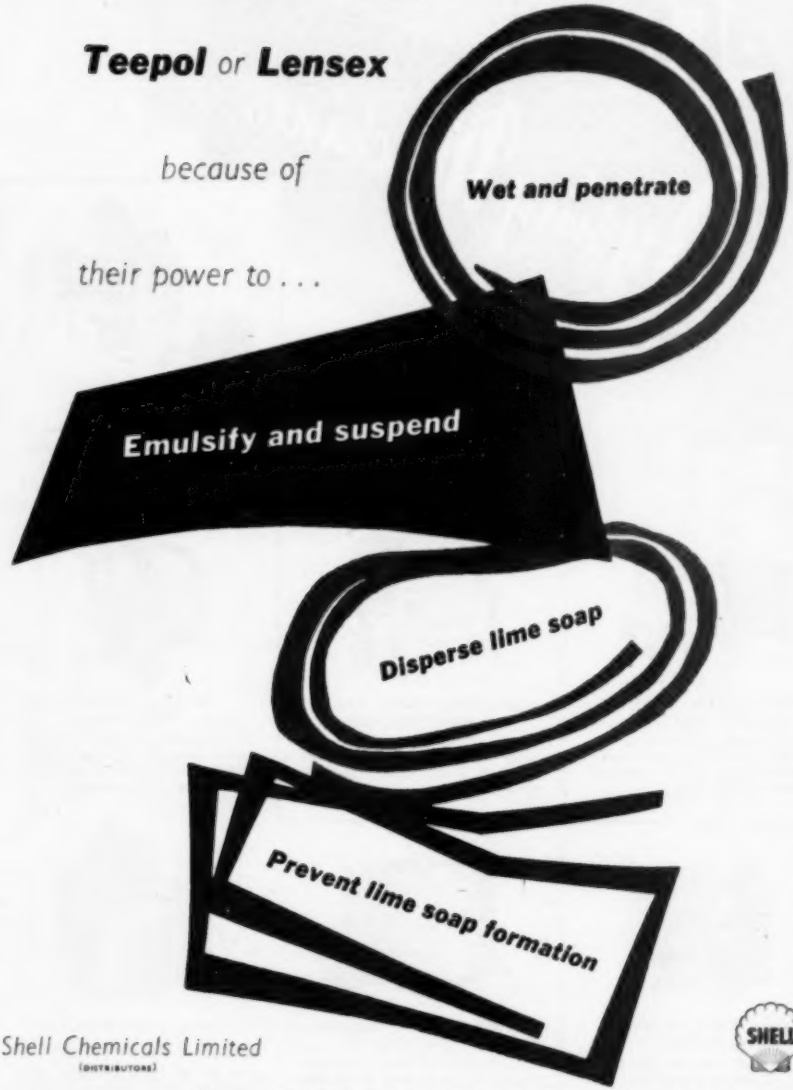

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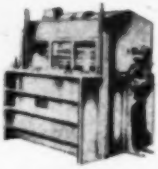

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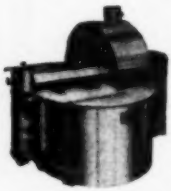

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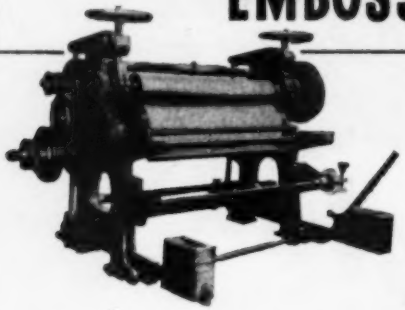

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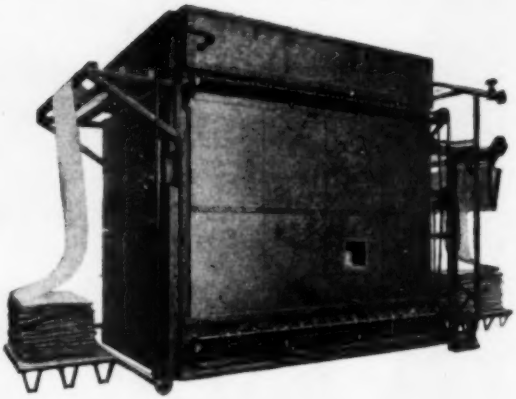

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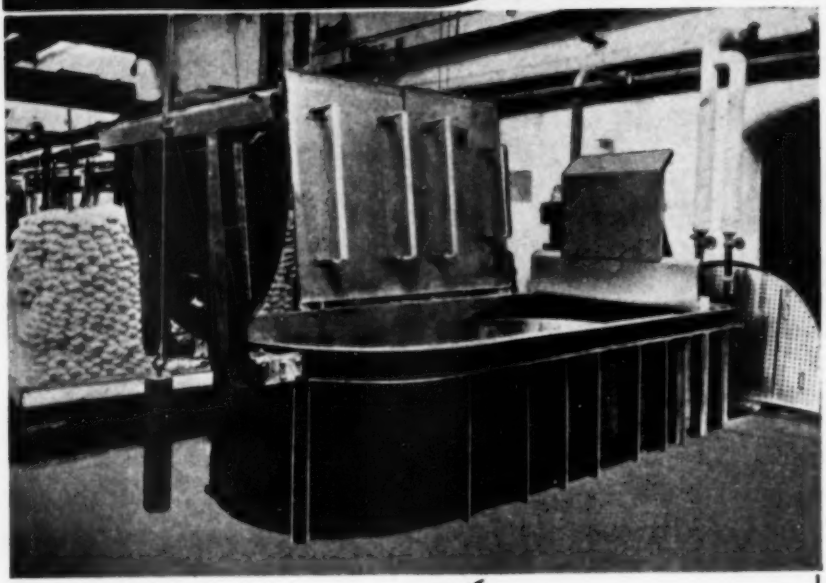

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1952 and pages 237–240 of the July 1952 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Place of the Technologist in our Industry — *F. L. Goodall*

Turkey Red Dyeing in Scotland—its Heyday and Decline — *R. A. Peel*

Non-felting Wool and Wool Mixtures (Mercer Lecture) — *F. C. Wood*

COMMUNICATIONS

New Methods of Analysis for Dyes and Intermediates — *C. H. Giles* et al.

Combination of Wool with Acids — *B. Olofsson*

Some Aspects of the Drying and Heating of Textiles—VII—The Stability of Thermal Modifications of Fibres in Subsequent Treatments — *J. M. Preston and S. P. Gundavda*

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Forthcoming Meetings of the Society (see also page xxix)

MANCHESTER SECTION

Unless otherwise stated meetings are held in the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m.

1952	
Friday 21st Nov.	LADIES' EVENING. Manchester College of Technology, commencing at 7 p.m. *Penny Plain. Twopence Coloured.* Lectures and demonstrations on the miscellaneous applications of dyestuffs. Speakers to be announced later
Friday 19th Dec.	Lecture to be announced later. Details to be announced later
1953	
Friday 16th Jan.	G. G. Taylor, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of Colour*
Friday 20th Feb.	W. Speke, Esq., B.Sc. (I.C.I. Ltd., Dyestuffs Div.). *High Temperature Dyeing of Viscose Rayon*
Friday 13th March	Manchester College of Technology. Afternoon and Evening Symposium on *New Fibres.* Details to be announced later
Friday 20th March	Joint meeting with the British Association of Managers of Textile Works. Midland Hotel, Manchester. Lecture by Dr. Fargher (Shirley Institute). Details to be announced later
Friday 17th April	ANNUAL GENERAL MEETING. Details of lecture to be announced later

NORTHERN IRELAND SECTION

All Meetings to be held in Queen's Hotel, Belfast at 7.30 p.m.

1952	
Wednesday 10th Dec.	A. S. Cluley, Esq. (Courtaulds Ltd.). *Some Observations on Practice in the Dyeing and Finishing of Rayon Materials* (Joint meeting with Textile Institute)
1953	
Wednesday 14th Jan.	E. R. Wiltshire, Esq. (I.C.I. Ltd.). *The Control of Azoic Dyeing Processes* (Joint meeting with Foremen Dyers Guild)
Wednesday 11th Feb.	F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.). *Pressure Dyeing and Bleaching with Possible Indication of New Developments*
Wednesday 11th March	Dr. B. C. Gee. *The Educational Side of Textile Processing*
March/April	ANNUAL GENERAL MEETING and DINNER (date will be announced later)

LONDON SECTION

All meetings held at 6 p.m. in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, unless otherwise stated

1952	
Friday 5th Dec.	Dr. P. R. Peacock (Royal Beatson Memorial Hospital, Glasgow). *Food Dyes*
1953	
Friday 9th Jan.	*Uses of Microscopy in Textile Dyeing and Finishing* (Clayton Aniline Co. Ltd.).
Friday 6th Feb.	F. Ward, Esq. (Courtaulds Ltd.). *Methods and Principles of Textile Fireproofing*
Friday 6th March	Title to be announced later

MIDLANDS SECTION

Except when otherwise stated, Lecture Meetings commence at 7 p.m.

1952	
Saturday 22nd Nov.	LADIES' EVENING — DINNER DANCE Black Boy Hotel, Nottingham
Wednesday 17th Dec.	*Colloquium.* Short papers on practical problems given by Local Members. King's Head Hotel, Loughborough

Midlands Section — *continued*

1953	
Wednesday 28th Jan.	J. Boulton, Esq., M.Sc.(Tech.), F.R.I.C., F.T.I. *Modern Dyeing Theory for the Plain Man.* Masonic Hall, Hinckley, 7.30 p.m. (Joint meeting with the Textile Institute and Hinckley Textile Society)
Thursday 19th Feb.	E. M. Walker, Esq., F.T.I., F.R.M.S. *The Chemist and Technologist in the Hosiery and Knitted Goods Industry.* (Joint meeting with the Textile Institute). Carpet Trades Canteen, Kidderminster, 7.30 p.m.
Wednesday 25th Feb.	E. R. Wiltshire, Esq., B.Sc. *Azoic and other Fast Dyes on Knitted Cotton Fabric.* College of Technology, Leicester
Friday 13th March	MIDLANDS SECTION DINNER. Royal Hotel, Leicester
Wednesday 18th March	G. H. Osborn, Esq., F.R.I.C., A.M.Inst. M.M. *Colour in Chemistry* (Joint meeting with the British Association of Chemists). School of Arts and Crafts, Derby
Wednesday 22nd April	ANNUAL GENERAL MEETING OF THE SECTION. Gas Board Theatre, Nottingham

Towards the end of the session Dr. H. White (Textile Research Institute, Princeton, U.S.A.) will lecture in Nottingham. Particulars later

HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Huddersfield, at 7.30 p.m., unless otherwise stated

1952	
Tuesday 2nd Dec.	Sir Wallace Akers, F.R.S. *Chemistry and the Textile Industry* (Joint with R.I.C. Huddersfield Section)
Tuesday 16th Dec.	Professor J. B. Speakman. Title later
1953	
Tuesday 20th Jan.	G. H. Rostron, Esq., A.R.I.C. (J. Crosfield). *Textile Process Soaps*
Tuesday 17th Feb.	INTER-SECTIONAL DISCUSSION
Tuesday 17th March	Dr. H. A. Thomas (Courtaulds). *The Rôle of Dyeing, Printing, and Finishing in Fabric Development*
Tuesday 7th April	ANNUAL GENERAL MEETING followed by a lecture — Dr. J. F. Gaunt. *A Study of the Afterchrome Process of Wool Dyeing*

SCOTTISH SECTION

All meetings at St. Enoch Hotel, Glasgow, 7 p.m., unless otherwise stated

1952	
Tuesday 9th Dec.	Ministry of Fuel and Power. *Steam Utilisation and Boiler Efficiency*
1953	
Tuesday 13th Jan.	J. C. Brown Esq. (Clayton Aniline Co. Ltd.) *Microscopy for the Dyer and Finisher*
Tuesday 17th Feb.	J. Barr Esq. (Turnbull's Ltd.) Details later
Tuesday 17th Mar.	ANNUAL GENERAL MEETING, to be held at the ST. ENOCH HOTEL, GLASGOW at 7 P.M. To be followed by *Brains Trust* at 7.30 p.m. The Panel is to be formed from Sectional Members and details will be circularised later. By courtesy of the College Authorities an invitation has been extended to inspect the new laboratories on this occasion

MANCHESTER JUNIOR BRANCH

All meetings commence at 6.30 p.m. in the Reynolds' Hall, College of Technology, Manchester.

1952	
Monday 1st Dec.	*The Dyeing of "Ardil" Non-Wool Unions* L. Biggens Esq.

Continued on page xxix

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THE JOURNAL

OF THE

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Volume 68—Number 11 NOVEMBER 1952 *Issued Monthly*

Proceedings of the Society

Colour Photography

R. B. Collins and C. H. Giles

Meetings of the Huddersfield Section held in Field's Café, Huddersfield, on 23rd November 1948, Mr. D. Hanson in the chair; of the London Section held in the rooms of the Royal Society, Burlington House, Piccadilly, London, on 6th January 1950, Mr. H. à Brassard in the chair; and of the Leeds Junior Branch held in the University, Leeds, on 27th November 1951, Mr. C. L. Bird in the chair

This is a review of the physical and chemical principles of colour photography and of its historical development. The subjects covered are indicated by the section headings—

I—History

1. Introduction
2. The Photographic Process
3. Primary Colours
4. Maxwell's Experiment
5. Duhauron
6. Cros
7. The "One-shot" Camera
8. The Mosaic-screen Plate
9. Dye Sensitisation
10. Progress with the Mosaic-screen Plate
11. The isoCyanines
12. Commercial Screen Plates
13. Dufaycolor
14. Colour Prints, etc.
15. Tripacks, Monopacks, and Colour Development
16. Sensitisers for Monopacks
17. Kodachrome and Agfacolor
18. Colour Cinematography
19. Summary of Present-day Practice

II—General Principles

(A) Colour Vision and Primary Colours
1. Colour Vision
2. Additive Primary Colours
3. Subtractive Primary Colours

(B) Basic Principles of Colour Reproduction
1. Additive Synthesis
2. Subtractive Synthesis

(C) Additive Processes
1. Simultaneous Projection Processes
2. Successive Projection Processes
3. Processes based on Optical Colour Separation
4. Mosaic Screen Processes

(D) Subtractive Processes
1. Separation Negative Processes
2. Monopack Processes

(E) Colour Cameras
1. The Still Camera
2. The Technicolor Camera for Motion Pictures

(F) Colorimetric Principles
1. The Trichromatic System
2. The Monochromatic System
3. The Chromaticity Diagram
4. Colour Reproduction by Colour Photography
5. Colour Temperature
6. Duplication of Colour Photographs
7. The Problem of Sound Reproduction in Colour Cinematography

III—The Chemistry of Colour Image Formation

1. Chemical Toning Processes
2. Dye Toning Processes
3. Silver–Dye-bleach Processes
4. Dye-bleach Process
5. Colour Development Processes
6. Tanning Processes
7. Colloidal Silver Process

IV—Conclusion

Appendixes

A. Commercial Name Index
B. Bibliography and Literature Review

I—History

1. INTRODUCTION

Colour photography has almost completed a century of existence. Clerk Maxwell, one of the great mathematical physicists of the Victorian era, first published a suggestion for a practicable process in 1855, and demonstrated it successfully to an audience at the Royal Institution in 1861. The method he used was cumbersome, and nearly fifty years elapsed before technical improvements enabled a simple commercial process to be offered to the public, but in the second half-century of the history of this now important branch of technology, very great advances have been made. Colour photography has not attracted such widespread popular attention as have many other modern applications of science and invention; yet it must surely rank as one of the outstanding technological achievements of modern times. It is now one of the specialised branches of dye and fine-chemical technology, and its use in the motion picture industry consumes significant quantities of intermediates and colouring matters. Moreover, colour photography has applications in medicine, in engineering, and in numerous branches of scientific and technological research.

The present review is an attempt to fill a gap in the literature by a full survey, in small compass, of the physical and chemical principles of colour photography, significant new advances being shown in their historical setting against the background of the development of the subject as a whole. The literature cited represents but a small fraction of the relevant material stored in the archives of patent offices and libraries, but it should be sufficient as a guide to those who wish to pursue the subject further.

Modern colour photographic methods depend upon the use of many physical and chemical principles, and for the convenience of the reader it seems better to place descriptions of such principles together under the appropriate headings, even though this involves splitting up the descriptions of certain important processes between different sections of the review.

2. THE PHOTOGRAPHIC PROCESS

The history of photography dates back at least to 1839, when L. Daguerre in France and W. H. Fox Talbot in England independently announced practical processes for producing pictures of natural objects by the action of light on surfaces sensitised with silver salts. Daguerre's process (*daguerreotype*) was the more elegant and attracted the greater attention at the time, but the positives he produced were made on a metal plate and could not be reproduced. Fox Talbot's process (*calotype*), although giving less delicate detail, was the true starting point for the development of modern photography. His method of preparing a paper negative, and from this as many paper positives as desired, was improved by Scott Archer's invention of the glass negative, with a wet collodion emulsion (1850), and later by the invention of the gelatin dry plate (Maddox 1871) and the celluloid film (Eastman 1889).

3. PRIMARY COLOURS

Colour-photographic processes are based on the principle of primary colours and the three-colour theory of vision, both of which have a long history. It is said [51] that the ancient Greeks taught that all colours could be compounded by correctly blending white, blue, red, and yellow; and in the early years of modern scientific development a similar hypothesis was adopted by several writers, e.g. Leonardo da Vinci (1452–1519). Le Blon, as early as 1722, experimented with full colour printing in mezzotint from three copper plates, one in blue, one in red, and one in yellow, with a fourth in black to deepen the shadows. While sound in principle and, indeed, exactly corresponding to the modern process, the method was tedious and uncertain, for without photography the relative proportions of the colours had to be decided entirely by the individual judgment of the operator.

Such early work was largely based on empirical ideas, but more exact knowledge was possible after Newton had announced his discovery of the solar spectrum (1672), which established the foundation of all subsequent scientific investigation of colour. A consideration of the empirical facts of colour reproduction by mixing three primaries led Thomas Young to propound* his famous three-colour theory of vision in 1802 *. This theory, which formed the basis on which colour photography was first attempted and which has largely guided its development, has been the dominating concept behind work on colour vision ever since, though its exact validity is still the subject of some controversy. It supposes that the eye perceives colour by three distinct types of nerve receptor in the retina, sensitive to red, green, and blue or blue-violet light respectively (§ II A 1).

4. MAXWELL'S EXPERIMENT

In 1855, Clerk Maxwell [101], who was then only twenty-four years old, made the first suggestion for a process of recording photographs in full colour in these words —

> [Young's] theory of colour may be illustrated by a supposed case taken from the art of photography. Let it be required to ascertain the colours of a landscape by means of impressions taken on a preparation equally sensitive to rays of every colour. Let a plate of red glass be placed before the camera, and an impression taken. The positive of this will be transparent wherever the red light has been abundant in the landscape, and opaque wherever it has been wanting. Let it now be put in a magic lantern along with the red glass, and a red picture will be thrown on the screen. Let this operation be repeated with a green and a violet glass, and by means of three magic lanterns let the three images be superimposed on the screen. The colour on any point on the screen will then depend on that of the corresponding point of the landscape, and by properly adjusting the intensities of the lights, etc., a complete copy of the landscape, as far as visible colour is concerned, will be thrown on the screen. The only apparent difference will be that the copy will be more subdued, or less pure in tint than the original.

* It is said [102] that Wunsch anticipated Young by ten years.

This description* (cf. Fig. 7) could hardly be bettered to-day.

On 17th May 1861 Maxwell [102] followed up this suggestion by demonstrating before a gathering at the Royal Institution in London the historic experiment in colour photography which has so often been described. As a Scotsman, he characteristically chose for his coloured object a tartan pattern, in the form of a silk bow, placed on a background of black velvet. This he photographed through three filters as he had himself suggested earlier, but for some reason which has not been explained he exposed a fourth plate also, behind a yellow glass filter. The red, violet, and green exposures were made through plate glass cells holding solutions of ferric thiocyanate, cuprammonium sulphate, and cupric chloride respectively. The collodion plates then available had extremely low sensitivity to any colours but the violet and blue, and Maxwell was obliged to give very long exposures through all except the blue filter†, and the original report of the demonstration records that "a coloured image was seen, which, if the red and green images had been as fully photographed as the blue, would have been a truly-coloured image of the ribbon" [102] ‡.

5. DUHAURON

Clerk Maxwell was a mathematical physicist whose interests lay in the theory of colour and colour vision. His experiment was carried out only to prove the truth of his own interpretation of Young's colour vision theory and not with the aim of advancing photographic practice, which indeed in those days was not considered a branch of science. He appears to have taken no further practical interest in the subject, but others soon began to follow it up, and of all subsequent inventors in this field, one name, that of the Frenchman, Louis Ducos Duhauron, is outstanding. During more than thirty years of great inventive activity, Duhauron was the first to describe at least four inventions—(*a*) the "one-shot" camera (§ I 7), (*b*) the mosaic-screen plate (§ I 8), (*c*) subtractive colour printing (§ II B 2), and (*d*) tripack emulsions (§ I 15)—one or other of which is employed in every colour-reproduction system in use to-day.

Duhauron§ published his work in a series of books and patents during 1869–1899 [103], but he wrote his original paper, in which he described the one-shot camera and the mosaic-screen plate, in 1862, at the age of twenty-five. He is said to have requested that it be presented to the Académie des Sciences, but this request was refused on the ground that he had no practical proof of the correctness of his suggestions. The paper was eventually incorporated with others in a book published in 1897 by his brother [103]. Duhauron's ideas were developed and brought to the stage of commercial success by others, mostly during 1905–1935. He himself died in 1920 in a state of great poverty.

6. CROS

Contemporaneously with Duhauron, another French inventor, C. Cros, independently published theoretical suggestions which were to exercise a great influence on the subsequent development of the subject. His publication of 1869 [103] described methods of taking three primary-colour records, including the use of filters and also the placing of a prism behind the lens to disperse the light so that the separate images could be formed by recording different portions of the spectrum. He described viewing instruments ("chromoscopes") involving mirror systems, the superposition of coloured impressions, and the synthesis of coloured images by persistence of vision in a zoetrope. This last idea received great attention at one period as a method of obtaining cinematograph pictures in colour (§ I 18(*a*)).

7. THE "ONE-SHOT" CAMERA

Duhauron devised a camera with a system of half-silvered and wholly silvered mirrors so arranged behind the lens that the light beam was equally divided between three plates, each being set behind one of the three primary-colour filters. Thus all three negatives could be taken with one exposure, and colour photography of moving objects would be possible. Maxwell's taking process could thus be very much simplified. The viewing process was simplified also in a somewhat similar, but later invention by Duhauron, in which the three positive transparencies were simultaneously viewed, through a single lens, behind their appropriate filters. One of his inventions described an instrument of this type which could be used for both taking and viewing the pictures.

8. THE MOSAIC-SCREEN PLATE

This invention adapted a principle which had been used by a school of French painters, known as *pointillistes*, who juxtaposed minute colour dots in place of areas of continuous tone. At a sufficient distance these appear to the eye to blend into a continuous tone, just as do the dots in half-tone illustrations now used in books and journals, though at that time this method of illustration had not been invented. Duhauron proposed to cover a sheet of paper or a glass plate with a series of alternating fine lines in the three primary colours. These were to be applied either by photography or by mechanical means. If the lines were correctly proportioned, when viewed at a distance they would appear a pale neutral grey. A red would be seen if the remaining two colours were blocked out, and a purple if the red and blue only were seen. By suitably blocking the three colours to different degrees, the screen could be made to appear any desired hue. It would not be necessary to do this by hand or mechanically, for "that which may be done by an expert hand may all be done by natural forces", wrote Duhauron, and he proceeded to explain how, using this principle, a coloured object would reproduce its colours in a photograph. The line

* It is interesting to note that colour television operates on these principles.

† 8 min., 12 min., 6 sec., and 2 min. for the red, green, blue, and yellow filters respectively. The specifications of the filters are not on record, but the exposure required without a filter was about 3 sec.

‡ A reproduction of this picture, made from the original negatives, is shown in a recent publication [40].

§ For some account of Duhauron's background and interests, see an interview in the *British Journal of Photography* and the obituary notice in the same Journal [1].

screen could, for example, be coated with a photographic emulsion which would give a direct positive print by light action*, the emulsion being exposed behind a camera lens, *through* the line screen.

Consider now the photography of a coloured object, say in blue, with such an arrangement. The object would form an image on the line screen, but only the blue lines would transmit its blue light through to the emulsion, which would be placed behind the screen. Consequently, on viewing the finished result after development to the positive, the blue lines over the area of the image would appear clear and the other two sets of lines would be blocked out. The image would thus reproduce the original colour of the object. The argument can be followed out similarly for any other colour, and for white or black, which will, respectively, affect the emulsion behind the whole screen or produce no action (cf. Fig. 5).

Duhauron's mosaic screen idea has remained to this day the simplest possible method of producing a colour photograph. It is the basis of the well known Dufaycolor process. At the time it was first conceived, however, the idea was impracticable, because it requires an emulsion which has at least similar sensitivity to red and green as to blue rays, and none such then existed.

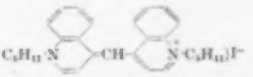
9. DYE SENSITISATION

Little advance could be made after Duhauron's and Cros's first disclosures until photographic emulsions, hitherto sensitive only to violet and blue, could be made sensitive to all parts of the spectrum. The necessary discovery, one of the most important in the whole history of photography, was made in 1873. Like so many classic scientific discoveries, it was made entirely by chance, when H. W. Vogel [151a] noted that some dry plates had an unexpected sensitivity to the green as well as to the blue portion of the spectrum. Vogel traced the cause of the increased sensitivity to the presence of a yellow dye which had been added to the emulsion to prevent halation†. Supposing that the dye had produced sensitivity to light which it itself absorbed, Vogel next examined a number of other colouring matters for their possible activity, and discovered that not all dyes acted as sensitisers, and that of those which did so, each sensitised to a particular region of the spectrum, some down even as far as the red. He noted the red-sensitising power of chlorophyll, and in 1876 Duhauron used this colouring matter to sensitise wet collodion plates for colour photography. Without the guidance of a scientific principle in selecting sensitisers, only the most empirical methods could be used in examining substances for their possible value in treating plates. Thus Cros in 1879 examined the sensitising activities of chlorophyll, a tincture of black currants preserved in brandy, an aqueous extract of marsh-mallow flowers, a tincture of carthamin, and an aqueous extract of bullock's blood [152].

* The principle of reversal development, now used, was discovered in 1868 by Russel [12].

† Halation is a fogging of the picture due to reflection of light, mainly from the plate.

. The violet-blue cyanine (*C.I.* 806) (Williams 1856)—



was found by Vogel in 1875 to be a reasonably good red sensitiser, and remained in use for this purpose until the *iso*cyanines were discovered nearly thirty years later. It had faults, e.g. it suffered from a tendency to fog the plate; and other dyes, e.g. Alizarin Blue S (*C.I.* 1067) and Coerulin (*C.I.* 783) were sometimes used instead. None of these products was, however, entirely satisfactory. Nevertheless, with the availability of even a partly successful process for preparing plates sensitive to the whole spectrum, colour photography began a further stage of its advance. Miethe in Germany and Ives in the U.S.A. made use of the new facilities, and produced successful colour pictures by Maxwell's three-colour separation process (ca. 1886), Ives designing also a triple-projection lantern and viewing-box (chromoscope).

10. PROGRESS WITH THE MOSAIC-SCREEN PLATE

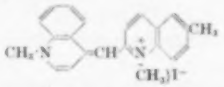
The mosaic screen process requires not only a fully colour-sensitive plate but also a fully colour-corrected lens, i.e. one which brings rays from the whole spectrum to the same focus. The discovery of new varieties of optical glass by Abbe and Schott, of Jena, in 1880 led to a great improvement in photographic lenses, and satisfactory colour correction became a possibility. Thus by the early 1890s Duhauron's process, so attractive in its simplicity of operation, had come within the bounds of feasibility. Several inventors began to explore its possibilities, and by the middle of the decade the Lumière brothers in France, Joly in Dublin, and McDonough in the U.S.A. had all commenced experiments upon it. Joly ruled the colour screen mechanically with a set of fine pens; he obtained satisfactory pictures before 1894 [74], and exhibited the process at the Royal Photographic Society's exhibition in 1898, while in 1893 the Lumière brothers had taken the first portrait of a living person in natural colours [6]. Nevertheless, colour plates could not yet be marketed, on account of the unsatisfactory keeping properties of the emulsions sensitised with the known dyes and the comparative coarseness of the screens produced up to that time.

During this same period, several novel processes depending on optical effects, such as diffraction and refraction, were invented, but these have been little more than laboratory curiosities. They are described later (§ II c 3).

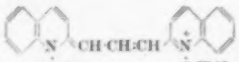
11. THE *ISO*CYANINES

Most of the basic principles of the present-day colour-photographic techniques had thus been worked out between 1860 and 1900, but no really successful process had been demonstrated. In the opening years of the present century, however, the final discovery was made which enabled colour photography to grow from a laboratory curiosity

to a commercially acceptable process. This was the synthesis of the isocyanines [56], an entirely new series of sensitising dyes for the orange and red, in which one of the quinoline nuclei of the cyanine type of dye is linked with the other at the 2- instead of the 4-position. They were discovered in the Hoechst laboratories of the firm of Meister, Lucius & Brüning, from 1901 onwards. One of the series, König's Ethyl Red (*C.I.* 807) —



was utilised by the firm of Otto Perutz for a "panchromatic" plate introduced commercially in 1903, but the first entirely satisfactory panchromatic sensitiser was Pinacyanol (*C.I.* 808), in which the intermediate chain is lengthened by one vinylene group—



This dye was discovered by König in the Hoechst laboratories in 1905, and used for panchromatic plates by the English firm of Wratten & Wainwright in 1906. There has been continuous progress since that date in this most important field of organic chemistry [e.g. 58, 75, 80, 105, 152], further discussion of which is outside the scope of this review.

12. COMMERCIAL SCREEN PLATES*

The stage was now set for the introduction of commercial additive colour-screen processes. The first type of screen produced commercially was the *irregular* mosaic screen introduced in 1907 by the Lumière brothers as the Autochrome plate. This screen was produced by dyeing starch grains red, green, and blue, and after thorough mixing, scattering them on plates previously coated with a gum layer. Any excess of grains not adhering to the gum layer were shaken off, and after rolling, the interstices between the grains were filled by dusting with carbon black, to avoid dilution of the colours with white light. After drying, the screen was covered with a protective varnish layer, and a panchromatic emulsion was coated thereon. This plate enabled any photographer to obtain good results with his normal equipment, even though the exposures required were lengthy (1 sec. at *f*/8 in summer sun; this is about fifty times the exposure needed with present-day colour films). From that time until the outbreak of the First World War there was much activity by inventors and manufacturers in producing new varieties of colour plate, and a considerable interest in colour photography was displayed by serious amateur photographers. Indeed, an immense number of patents describing varieties of screen plates continued to appear until recently. It is said that more than thirty varieties were either commercially in production or under development in 1910, though only a few were widely used [131]. The Autochrome plate was in use up to 1933, when it was replaced by mosaic-screen films (Filmcolor and Lumicolor).

The first *regular* mosaic screen was introduced in this country by Finlay in 1912 as the Paget plate. This consisted of regularly spaced ink lines, and was coated on glass without an emulsion. It was placed in the camera in contact with a panchromatic plate, and after exposure the latter was removed, developed, reversed, and bound up in register with the screen. The process has several advantages over those in which screen and plate are integrally combined. The Finlay process, and also the Agfacolor plate (coated with an irregular mosaic of dyed resin particles), introduced about the same period, were both in use up to a few years ago, and the Finlay plate has just been revived in this country as the Johnson Colour Screen Process. There were two other screen plates on the British market in 1914—the Dioptichrome and the Omnicolor.

13. DUFAYCOLOR

Dufay in France had introduced the Dioptichrome plate, in which the screen had printed lines of colour, in 1910, and after the first World War he continued to devote attention to its improvement and to the application of the screen to films. About 1927 an English firm of paper manufacturers, Spicer's Ltd., began to take an interest in its possibilities, and in co-operation with Ilford Ltd. carried out intensive research in order to perfect the design of the screen and to apply it to roll film by mass-production methods. They were successful, and their product, Dufaycolor film, introduced in 1934, probably represents the limit of achievement of the mosaic-screen process. It has a higher light transmission than any previous mosaic-screen plate or film, and the ruling of the exceedingly fine lines of the screen (or *réseau*) represents a considerable achievement in applying precision processing to mass production. The Dufaycolor film has intersecting regular lines of colour, mechanically ruled (Fig. 6) with a width as fine as one thou (0·001 in.), and transparencies can be projected by lantern without the pattern being perceptible at normal viewing distances from the screen. It has been adapted as a negative–positive process for cinematograph work, and was the first process which made colour photographs popular with the ordinary snapshot camera user. It is reliable in use and the colour rendering is good, but its popularity has been diminished by the advent of the monopack subtractive processes. Dufaycolor film has been marketed successively by Spicer's Ltd., Spicer-Dufay Ltd., Dufay-Chromex Ltd., and Dufay Ltd.

14. COLOUR PRINTS, ETC.

Fundamental discoveries in the technique of "tanning" (insolubilising) gelatin were recorded in the nineteenth century, and have been made the basis of successful methods of preparing colour prints on paper and coloured positive cinematograph films. The knowledge that gelatin containing a soluble dichromate becomes insolubilised if exposed to light is almost as old as photography,

* For an account of the history of screen-plate processes up to 1907, and for the methods of processing then used, see Brown [17].

but because of the lack of colour of the mixture, no use could be made of the action in photography until Poitevin in 1855 mixed carbon black with the emulsion and, by washing away the unaffected gelatin with hot water after exposure under a negative, obtained a black-and-white positive image. This came to be known as the "carbon" process, even though other insoluble pigments were subsequently used.

Four other important discoveries in this field were made at later dates [51]—

(*a*) Better results are obtained if the "carbon" image is transferred, face downwards, to another surface before washing off (Swan 1864).

(*b*) A silver image placed in contact with the dichromated gelatin will tan it equally as well as exposure to light (Manly 1898). This is the basis of the Carbro process.

(*c*) Certain dyes are adsorbed from solution by untanned, but not by tanned, gelatin (Edwards 1867).

(*d*) The gelatin of a normal silver emulsion is tanned in the image portions by developers not containing sulphite (Warnerke 1881).

A fuller description of the use of these discoveries in practical printing processes is given in a later section of this review (§ III 6).

Duhauron had described the principle of producing colour prints by superimposition of three transparent positives coloured with primary colours as early as 1869, and he is said to have been the first person to prepare a colour print on paper. In 1877 he exhibited in Paris colour prints prepared by the carbon process, and Cros used imbibition printing (§ III 6 (*c*)) for preparing colour prints in 1880.

Three-colour half-tone illustration on paper, in which the individual colour records are produced from three-colour separation negatives, was introduced about 1890.

The subtractive principle, on which the coloration of prints and tripack transparencies (§ I 15) is based, is described in § II B 2.

Besides the successful introduction of mosaic-screen plates, many other important advances were made in the early 1900s. For example, colour printing on paper was made available by the commercial introduction of the Pinatype process in 1905 and the three-colour carbon process in 1915. Ideas also began to take shape and inventions appeared which led eventually to the present-day monopack colour films.

A short-lived process for producing direct colour photographs in ordinary snapshot cameras was introduced in this country about 1929. It utilised a separable tripack (§ I 15), consisting of three individual films, for the primary records, and prints were made on paper. Owing to the lack of proper registration between the negatives, definition was poor. The colouring agents were probably inorganic salts [cf. 147].

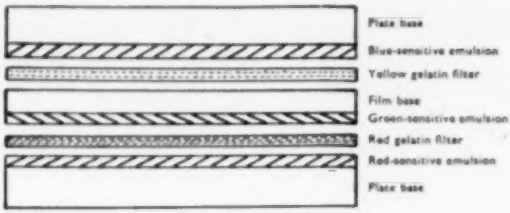


FIG. 1—The First Tripack (Duhauron 1895)

15. TRIPACKS, MONOPACKS, AND COLOUR DEVELOPMENT

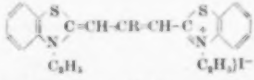
Duhauron in 1895 and 1897 had patented, and in a publication of 1899 had further described, a "selection of light rays by an alternation of colour filters and plates or sensitive films, formed like the leaves of a book or polyfolium, placed in the dark slide". The actual arrangement he latterly described is as shown in Fig. 1. After exposure, the plates and film were to be taken apart for individual development and printing in the appropriate colours. This tripack arrangement is bulky and, owing to the difference in position of the different emulsions, would give unsharp pictures, but it is fundamentally the arrangement now used in the majority of modern processes, except that considerable simplification has been introduced by coating all the layers in superposition on one support to ensure sharp register of the three records (the red filter is no longer used). This is an arrangement which was first proposed in 1905 by Schinzel [51], and is described as an *integral tripack* or a *monopack*.

Schinzel's arrangement could not be used at the time because it could not be made into a colour positive. Some method of transforming the silver images into images of the appropriate colours was required. A method by which such a transformation could occur, by formation of a dye image during development, simultaneously with the silver image (*colour development*), was first described by Homolka in 1907 [51]. Colourless *leuco* dyes were employed to develop the exposed silver image, and in so doing were themselves oxidised to the coloured forms. This process, however, is rather limited in its application, and Fischer and Siegrist [49] of the Neue Photographische Gesellschaft, Berlin, studied (1911–1914) another method of colour development based on the formation of indamine and indophenol dyes by sulphite-free developers. Gusserow and Anderson (1901) had noted the formation of such dyes when developers acted upon light-exposed dichromated gelatin; Fischer and Siegrist extended the reaction to exposed silver halide emulsions. Their paper (1914) is classic, and describes almost all reactions used in modern colour development, though their discoveries could not be applied for nearly twenty years. Fischer [48] in 1911 had actually proposed an integral tripack on Schinzel's principle, in which the appropriate colour-coupling agents were added to the emulsions before coating on the base, so that in a single development each layer would appear in its correct colour. This

method is simple and attractive, but owing to the impossibility at that time of ensuring that the coupling agents remained in their own layers without diffusion into the adjacent ones*, and to the fact that no suitable sensitisers were known, no further progress was then made. The outbreak of war prevented further advance along this promising line for nearly twenty years, and the use of mosaic-screen plates remained the only simple method for preparing colour transparencies.

16. SENSITISERS FOR MONOPACKS

In mosaic-screen processes, the particular regions of the spectrum to be separately recorded are determined by the filter dyes used, and an emulsion having more or less uniform sensitivity to all wavelengths is desired. In the monopack process, the only filter is a yellow one immediately below the top emulsion. Consequently both lower layers receive green and red light, and if they are each to record one of these, they must each be sensitised to that one waveband alone. Sensitisers suitable for this purpose were not available until after the First World War. Typical examples of such sensitisers are—



($R = H$) and the more effective β-methyl derivative ($R = CH_3$) (Hamer 1928).

A further difficulty encountered in sensitising monopack emulsions is the liability of the sensitisers to migrate to adjacent layers. The means by which this is prevented has not been disclosed. Further problems arise when additional reagents are added to the sensitised emulsions, e.g. colour couplers as in Agfacolor, or dispersing agents as in Kodacolor. These problems have necessitated extensive researches into the chemistry of sensitising dyes.

17. KODACHROME AND AGFACOLOR

About the year 1924 two amateur inventors in the U.S.A., Mannes and Godowsky, who had been experimenting with colour-photographic processes, became interested in Fischer's suggestions. By 1930 [99, 100] they had achieved success by a complex procedure which involved the separate processing of each layer in the monopack to its appropriate colour, the colour-coupling reagents being added to the developing solutions and not to the emulsions. By using different solutions to develop each layer and relying upon careful control of their depth of penetration, they were able to ensure that each one reached only its appropriate layer. Mannes and Godowsky† at about this time joined the Eastman Kodak firm and, with the facilities of this large research organisation available, progress was rapid. By 1935 the process was launched commercially as the Kodachrome film for substandard cinematography and 35-mm. still cameras. The development processes used were lengthy and highly complex, but the results produced were remarkably good and the film became an instant success.

Meanwhile the Agfa research laboratories [134] were also working on the monopack process, but they were adhering to Fischer's idea of incorporating the colour couplers in the emulsion layers during manufacture. To prevent wandering of the dyes they employed "anchoring" groups attached to the colour-coupler molecules. This device proved to be successful, and in 1936 the Agfacolor film based on this principle was first placed on the market. The developing process is much simpler than that used for Kodachrome.

Monopack processes have since been introduced by Ilford in Britain, by Gevaert in Belgium, and by Ansco in the U.S.A. Information regarding the various monopack films currently in use is summarised by Coote [20].

If the colour-coupling reagents are present in the film, development can be carried out by the photographer himself, and moreover, the film can be developed to a colour negative and positive prints in colour made from it as required. Agfacolor film was made available for a negative–positive process of this nature in Germany in 1939, and it was used for standard-size motion pictures during the recent war, and also for producing prints on paper (1943). Paper prints can be made by the Kodacolor process introduced in the U.S.A. by Eastman Kodak in 1942 (Fig. 4), which also incorporates the colour-couplers in the emulsions. The long-sought goal of inventors in colour photography, the production of paper prints in full colour by a direct printing procedure, has at last been realised.

Ektacolor (Kodak) represents a still further advance in the technique of these processes. This is a film incorporating coloured coupling agents for the self-masking process (§ II *v* 6(*b*)), and it also can be processed to a colour negative by the user.

These subtractive processes give greatly improved light transmission compared with the mosaic-screen additive process, the image is virtually grainless, and the colour rendering is very satisfactory. Hence they are very widely used*. It must be recognised that the availability of precision miniature cameras with high-grade lenses, which has enabled the cost per picture to be reduced to a reasonable level, has contributed very much to their success. The first modern precision camera, the Leica, was introduced in 1925, and it and other types were in use in large numbers by the time monopack colour films appeared in 1935.

18. COLOUR CINEMATOGRAPHY†

(*a*) *Additive (Colour-separation) Systems* [61]

Moving pictures shown by projection are an obviously suitable medium for experiments in colour photography, and at first sight Maxwell's original three-colour separation method appears to be ideal for this purpose, because the primary-colour positives may be presented to the eye in rapid succession by the projecting machine, and

* Fischer proposed tanning the gelatin emulsion before spreading, to prevent diffusion. This does not appear to have been a successful method.

† For an interesting biographical sketch of these two inventors, see Eastman [45].

* Mees [104] stated that in 1941 20,000,000 still pictures and about 75% of amateurs' motion pictures were made in colour.

† There were said to be eleven commercial processes operating in 1948 [112]. Only the better known ones are described in detail in this review.

they will be so blended by the persistence of vision as to give the appearance of a picture in full colours. It is not surprising, therefore, that the use of such a device was proposed very early in the history of moving pictures. (Cros, indeed, had suggested a somewhat similar principle in 1869, when he described the use of the zoetrope for giving coloured images (§ I 6).) Isensee in Germany in 1897 was the first to propose the use of the method with the cinematograph camera, and subsequently very many other inventors worked upon the idea. Isensee proposed that the three primary-colour filters should be placed in a disc rotated before the lens of the camera in synchronisation with the film, so that successive frames were exposed through each primary filter in turn. The film would then be processed normally to a black-and-white positive and shown in a projector equipped with another rotating set of filters identical with those used in exposing the film, each frame being projected through the primary filter through which it was taken.

In the next few years many inventors, including Friese-Greene, one of the originators of the cinematograph, turned their attention to this method. Smith, in 1906, using a two-colour process operating at 30 frames per second, was one of the first to bring it to success, and Urban and Smith launched it publicly two years later under the name of *Kinemacolor*. This was the first commercial colour cinematograph process. It created much interest at an exhibition in London that year, and again in 1911 when "farmyard and agricultural scenes, topical events, portrait studies, and a very moving picture play" were shown. In the same year there appeared a remarkable Kinemacolor film of the Delhi Durbar.

Nevertheless, in spite of this initial success, the rotating filter process failed to establish itself commercially. It has several inherent defects—viewing it for too long produces eyestrain; the pictures show "colour-fringing" effects with moving objects, because the images change their position between the successive colour frames; and high-speed cameras and projectors are required, causing considerably increased wear and tear on the films.

Much ingenuity was expended in attempts to overcome these difficulties. Thus some inventors exposed and projected the three records simultaneously. In the Gaumont process (1912) this was done by using three contiguous lenses in both camera and projector. Parallax errors are, however, introduced in this way. They can be eliminated only by using a single lens having a beam-splitting device behind it, but to split the light into three beams in a camera and to recombine the beams in the projector is a difficult problem requiring complex prismatic systems. Between 1915 and 1921 another solution of the problem was seen in a process called *Cinechrome*, which was commercialised with some success, and used a double-width film having the two series of colour records running down it side by side.

Processes have also been devised which not only use anti-parallax beam-splitting devices behind the lenses of both camera and projector, but which also include printing the three records in so small a size that they occupy little more space on the film than one normal frame. This inevitably results in a loss of definition because of the increased magnification necessary. The Bassani and Francita systems were worked on this principle, which has been revived recently in France in processes named *Rouxcolor* and *Dugromacolor* [118].

(*b*) *The Lenticular Process*

In 1928 the Kodak organisation introduced the Kodacolor* film for 16-mm. cinematography†. This worked on the lenticular process (§ II c 3(*d*)) employing a triple filter on the lens and parallel microcylindrical ridges on the film surface. The process was no longer used after Kodachrome was introduced. The original invention was patented by Berthon in 1909, and from about 1925 onwards a great deal of investigation was made toward its improvement, especially by the Keller-Dorian Corpn. and Eastman Kodak in the U.S.A. and by the firms of Siemens & Halske, Perutz, and the I.G. in Germany, but no ultimate success has been achieved, one major difficulty being the impossibility of producing duplicates satisfactorily.

(*c*) *The Dufaycolor Film*

The only mosaic-screen process to have been applied to commercial motion picture work is Dufaycolor. Dufaycolor film was used for a number of short professional films and for a film of the coronation scenes in 1937, which caused much interest at the time. While the colour reproduction is good, the loss of 75% of the illumination through the mosaic screen or *réseau* is a drawback and may account for this process not having been more widely used.

(*d*) *The Technicolor Process* [81]

A type of negative for motion pictures in which two films, carrying emulsions sensitised to different portions of the spectrum, are placed face to face and run simultaneously through the camera, had been patented in Germany in 1910. This arrangement is known as a *bipack*. After exposure, the two films are separated for individual development and printing. This invention avoids the difficulties associated with either rotating-filter devices on the camera or the presence of a mosaic screen on the film.

If the idea embodied in the bipack invention could be employed for making and projecting the positives, the combined processes would offer in theory a really satisfactory method of colour cinematography. Actually, Wall had already in 1906 suggested that the method of producing a coloured positive by transferring a dye, from a tanned image in gelatin, to clear gelatin on another film support, known as *imbibition printing*, might be used for printing cinematograph films in colour (§ III 6 (*c*) i). This process is the required complement of the bipack negative, for the two positives can be brought together in superposition on one film, which may then be projected in normal

* The present film of the same name operates on a different principle.

† This process was also used on the Continent for still pictures in miniature cameras at about the same time.

apparatus. It would, however, be anticipated that great difficulty would be encountered in obtaining true registration of the images in printing. Further, the bipack negative idea cannot be extended to a three-colour process for use in a normal camera, because of the impossibility of getting satisfactory register of the images in different planes, and use of a one-shot camera is essential. The design and operation of such a camera for motion picture work is again a matter of the greatest difficulty. Nevertheless, all these difficulties have been overcome, and the colour process most in use to-day for 35-mm. motion pictures, *Technicolor*, uses the one-shot camera and imbibition printing (§ II E 2).

In 1915 Kalmus, Comstock & Westcott Inc., a firm of engineers in Boston, U.S.A., began work on colour cinematography under the name of the Technicolor Motion Picture Corpn. Their first attempts were made with a two-colour additive process, employing a beam-splitting device behind the lens. The films were processed by tanning development and dye coloration (§ III 6(*a*)), the two colour positives being cemented back-to-back. Later they began to print their films by imbibition and to produce sound films in colour; and in 1932 they first employed a three-colour process. Since that time the output of Technicolor films has steadily risen, and the process has become well established in popular appeal. Much research has been undertaken to perfect the details of the complex processes involved, especially the transfer printing of the three colour records in accurate register.

(*e*) *Monopack Processes*

The Technicolor process is most satisfactory for commercial work because of its ability to produce unlimited numbers of positive prints of uniformly high quality, without the expense of using multi-layer film with silver-bearing emulsions. The printing procedure has been brought to a high state of efficiency, and plant is available for making prints on a large scale. The Technicolor camera, however, is a very cumbersome and expensive machine, and is unsuitable for many purposes. Monopack films as negative material offer several advantages, because they can be used with normal cameras, and so negative-positive monopack systems, e.g. Agfacolor, are being increasingly used. One interesting development has been to use a monopack negative material and to produce prints by the Technicolor method. The very successful film of the royal visit to Canada in 1951 was photographed on monopack film stock.

(*f*) *Two-colour Processes*

It is possible to obtain some measure of colour reproduction with a two-colour process, particularly in cinematography, where the considerable simplification of equipment effected by using two colours only has stimulated much work in past years. Duhauron, in 1897, was one of the first to suggest the possibility of a two-colour process. Later workers found that the best two-colour primaries are blue-green and orange-red, so chosen that in superposition a reasonably good approach to white is obtained. Violets, blues, and deep reds are absent in the prints, but flesh tints can be well reproduced. The effect on the eye is better than might be expected, partly because the imagination supplies qualities of colour which are not actually present. The great success of three-colour processes in recent years has now reduced the importance of two-colour methods, though, because of the much simpler equipment and processing required, they are still in use in commercial cinematography.

19. SUMMARY OF PRESENT-DAY PRACTICE

There are now three principal processes in use for still photography and three for motion pictures, as follows—

(*a*) *Still Photography*

(i) SEPARATION NEGATIVE PROCESS—Three negatives are exposed through the three additive primary filters, either simultaneously, in a specially designed camera, or in succession, in a normal camera. Positives are printed from each negative in the appropriate complementary colour and superposed for viewing.

(ii) MOSAIC SCREEN PROCESS—The film or plate is exposed, and the positive is also viewed, through a coloured mosaic screen.

(iii) THE MONOPACK PROCESS—Three emulsions are coated on one film base, and the layers respond severally to each of the three additive primary colours. A single exposure is given, and there are two general methods of processing—(*α*) Each layer is reversal-colour-developed to an image in the appropriate complementary colour, and the whole is viewed as a positive transparency. (*β*) Each layer is developed to a colour negative, which is used to reproduce positive prints either as transparencies or on paper.

(*b*) *Motion Pictures*

(i) THE MONOPACK PROCESS (as described under (*a*) (iii), above).

(ii) THE TECHNICOLOR PROCESS—A one-shot camera with a specially designed combined lens and prism system is used to expose three films simultaneously. These are developed to black-and-white negatives, which are used in a transfer process to print the three appropriate colour images in superposition on a single film base.

(iii) Combination of (i) and (ii), in which the negative is taken on monopack film and prints are made by three-colour imbibition printing as for Technicolor.

II—General Principles

A—Colour Vision and Primary Colours

1. COLOUR VISION

The principles of colour photography depend ultimately on the mechanism of colour perception by the eye, and brief reference must therefore be made to this subject. For further information, the sources mentioned in Appendix B should be consulted *e.g.* 61, 62, 159.

The six or seven distinct hues immediately recognisable when the spectrum is viewed as a

whole may be increased to over a hundred if only a narrow band is seen at a time, and to over a million different colour sensations by admixture of white and by altering the intensity of illumination. Yet all these varieties of colour can be exactly or closely matched by mixtures of only three primaries. This simple fact led Thomas Young to propound the three-colour theory of vision, an idea which has largely dominated a century and a half of research into human colour perception; yet the exact means by which the eye discriminates between light of different wavelengths and perceives "colour" is still incompletely understood.

The three-colour theory postulates three types of light-sensitive receptors in the eye, responsive to sections of the spectrum centred on the red, green, and blue regions respectively. Their responses must overlap to some extent to account for the continuous gradation of colours in the spectrum, and white light must stimulate them all. It has long been known that *two* general types of light-sensitive nerve terminals are readily detectable in the retina, known as *rods* and *cones*. The former are responsible for vision in dim light and the cones operate only in bright light*. Colours are distinguished only by the cones, and thus if Young's theory is correct, three varieties of cone should be identifiable; yet until recently no direct evidence was available that there is more than one variety present. In the last few years, however, Granit [64] has obtained positive evidence that receptors responsive to different spectral regions are present in the retina of various animal species. The technique used is a new one, in which electrical responses in the fibres comprising the optic nerve, evoked by light falling on the retina, are measured by means of microelectrodes held in contact with individual nerve fibres selected at random. Response curves have thus been found in about seven fairly sharply defined regions of the spectrum, the actual maxima varying from experiment to experiment, according to the particular nerve contacted. Not all the maximal responses are obtained from the eye of any one species; some species show only two, others four or five. There are also found some broad non-selective responses, which may be responsible for the sensation of white.

Objective tests of this nature can be made only on animal eyes; the human eye has to be investigated by subjective means, and a great number of suitable techniques have been developed. These include—experiments in matching narrow portions of the spectrum with three primary colours; determination of just perceptible differences of hue, saturation, etc. in different regions of the spectrum; variation of sensitivity to different colours after initial stimulation of the eye with white or coloured light; examination of sensitivity of limited areas of the retina with minute pencils of light; tests on different types of colour-blind individual; and many others. There is, however, in spite of the volume and variety of investigation that have been brought to bear on this problem, still no universally accepted theory of the mechanism of colour vision. Thus, for example, Wright [159] maintains a belief in the classical three-colour theory, with maximal responses at about 5800, 5460, and 4500 A.; Willmer [158] suggests a modified three-colour theory involving only one variety of cone receptor and two varieties of rods; and Hartridge [62] suggests that there may be as many as seven different receptor systems with a total of eight response maxima in the human eye. These last are the classical orange–red, green, and blue–violet, and five subsidiary maxima (deep red, red, yellow, blue–green, blue), whose influence decreases as the part of the retina containing the greatest number of cones (the *fovea centralis*) is approached.

Even so, whatever the real mechanism may be, it remains a fact that the fundamental principles of colour photography can be worked out intelligibly upon the classical theory, and this will be assumed correct for the purposes of the present review.

* There are about 65 million cones and 120 million rods in the human eye. These connect with about 800,000 channels in the optic nerve [65].

2. ADDITIVE PRIMARY COLOURS

It is found empirically that the three broad bands of light obtained by dividing the spectrum of white light into three parts, viz. red, green, and blue, when recombined produce white. These three bands of colour are known as *additive primaries* (Fig. 2A and 3A).

For most practical purposes it is true to say that all visible colours can be reproduced by a suitable mixture of these lights. If mixed, they must, however, be viewed in such a way that their effects upon the eye are added together. They may, for example, be shone simultaneously upon the same area of a screen (Fig. 2A), or repeatedly passed in very rapid succession across the field of view by a mechanical device. Transparent materials coloured with these primaries cannot satisfactorily be used superimposed, because no two of them have any common transmission band (Fig. 3A), and when placed in series in a beam they therefore absorb all the light.

3. SUBTRACTIVE PRIMARY COLOURS

In everyday circumstances, however, when colours are mixed, e.g. in dyed fabrics, as paints, or in colour-photographic layers, the hue seen is that remaining after each colouring matter has subtracted part of the incident light*. Under these conditions, if "primaries" are required for producing a full range of hues, then it is necessary to use the *secondary colours*, better known as *subtractive primaries*, which are the three colours obtained by subtracting red, green, and blue respectively from white light. They are also known as *minus primaries*, because they can be represented thus (Fig. 3B)—

White − red = Blue + green = Cyan (blue-green)
White − green = Blue + red = Magenta
White − blue = Green + red = Yellow

The effect of mixing these subtractive colours is shown in Fig. 2B.

The peculiar value of the subtractive primaries lies in the fact that, in contradistinction to the

* It is often assumed that three-colour half-tone book illustrations which are produced photographically are viewed thus. This is not strictly true, as careful inspection will show that their series of dots are partly superposed and partly side by side. Hence they are viewed partly subtractively and partly additively.

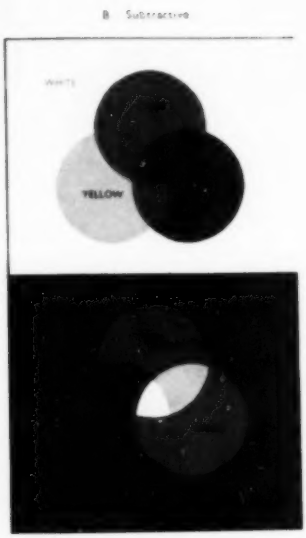


Fig. 2. Mixture of (a) Additive and (b) Subtractive Primary Colours

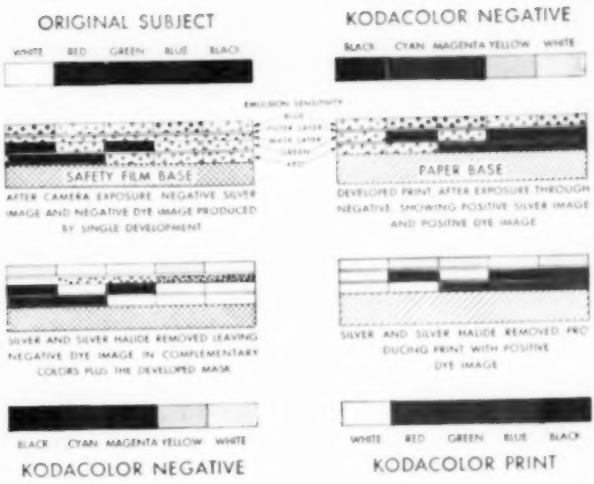

ORIGINAL SUBJECT
WHITE RED GREEN BLUE BLACK
KODACOLOR NEGATIVE
BLACK CYAN MAGENTA YELLOW WHITE
SAFETY FILM BASE
AFTER CAMERA EXPOSURE NEGATIVE SILVER IMAGE AND NEGATIVE DYE IMAGE PRODUCED BY SINGLE DEVELOPMENT
PAPER BASE
DEVELOPED PRINT AFTER EXPOSURE THROUGH NEGATIVE SHOWING POSITIVE SILVER IMAGE AND POSITIVE DYE IMAGE
SILVER AND SILVER HALIDE REMOVED LEAVING NEGATIVE DYE IMAGE IN COMPLEMENTARY COLORS PLUS THE DEVELOPED MASK
SILVER AND SILVER HALIDE REMOVED PRODUCING PRINT WITH POSITIVE DYE IMAGE
BLACK CYAN MAGENTA YELLOW WHITE
KODACOLOR NEGATIVE
WHITE RED GREEN BLUE BLACK
KODACOLOR PRINT

Fig. 4.—Principle of Monopack Negative-Positive Film

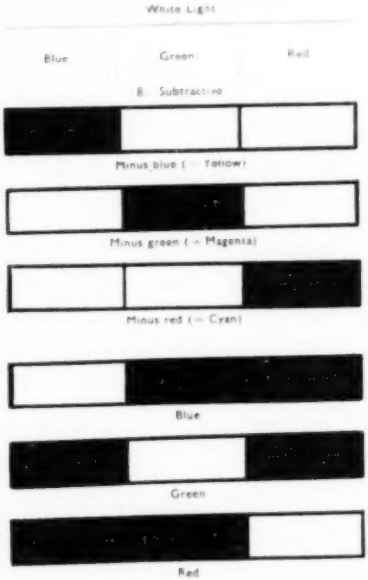


Fig. 3. Diagrammatic Illustration of the Transmission of (a) Additive and (b) Subtractive Primary Filters. When taken in pairs, (b) have common transmission bands while (a) have none

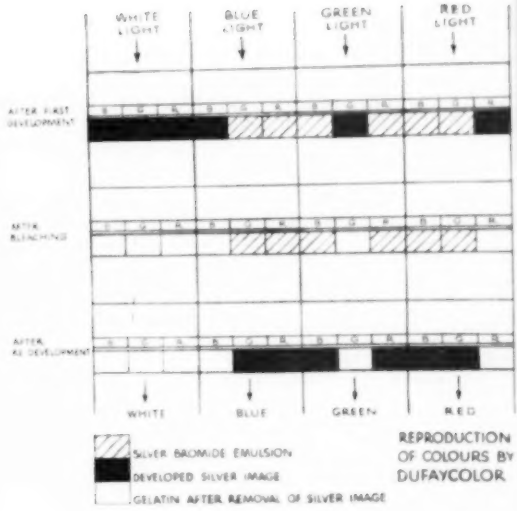


Fig. 5.—Dufaycolor Principle

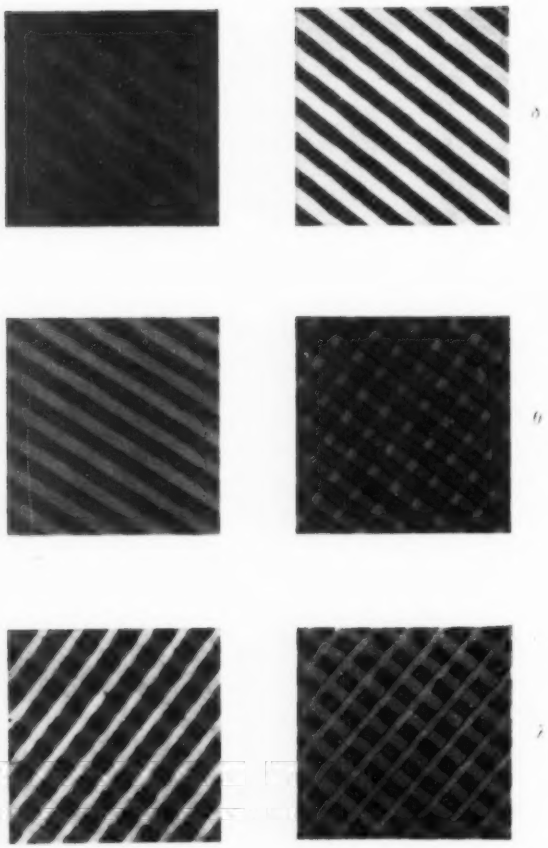


FIG. 6.—Stages in the Production of the Dufaycolor Reseau

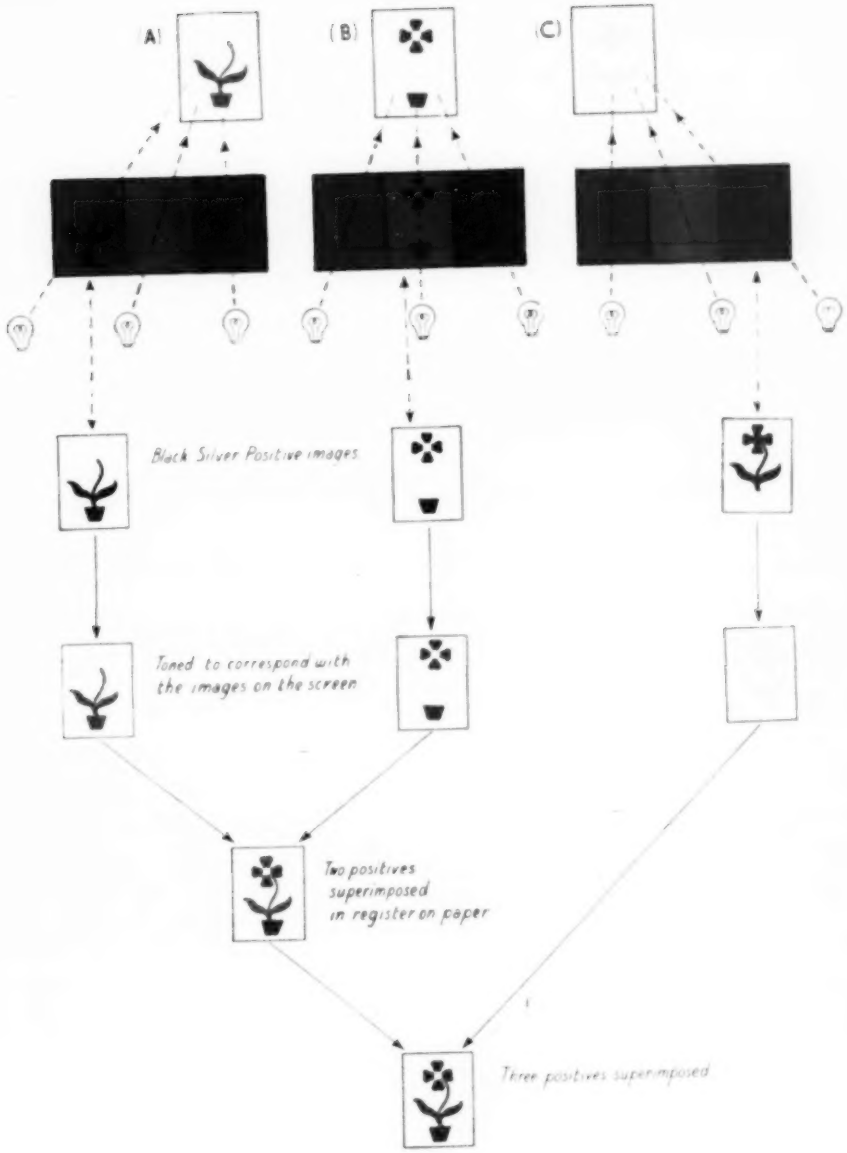


FIG. 2—Principles of Additive and Subtractive Three-colour Photography

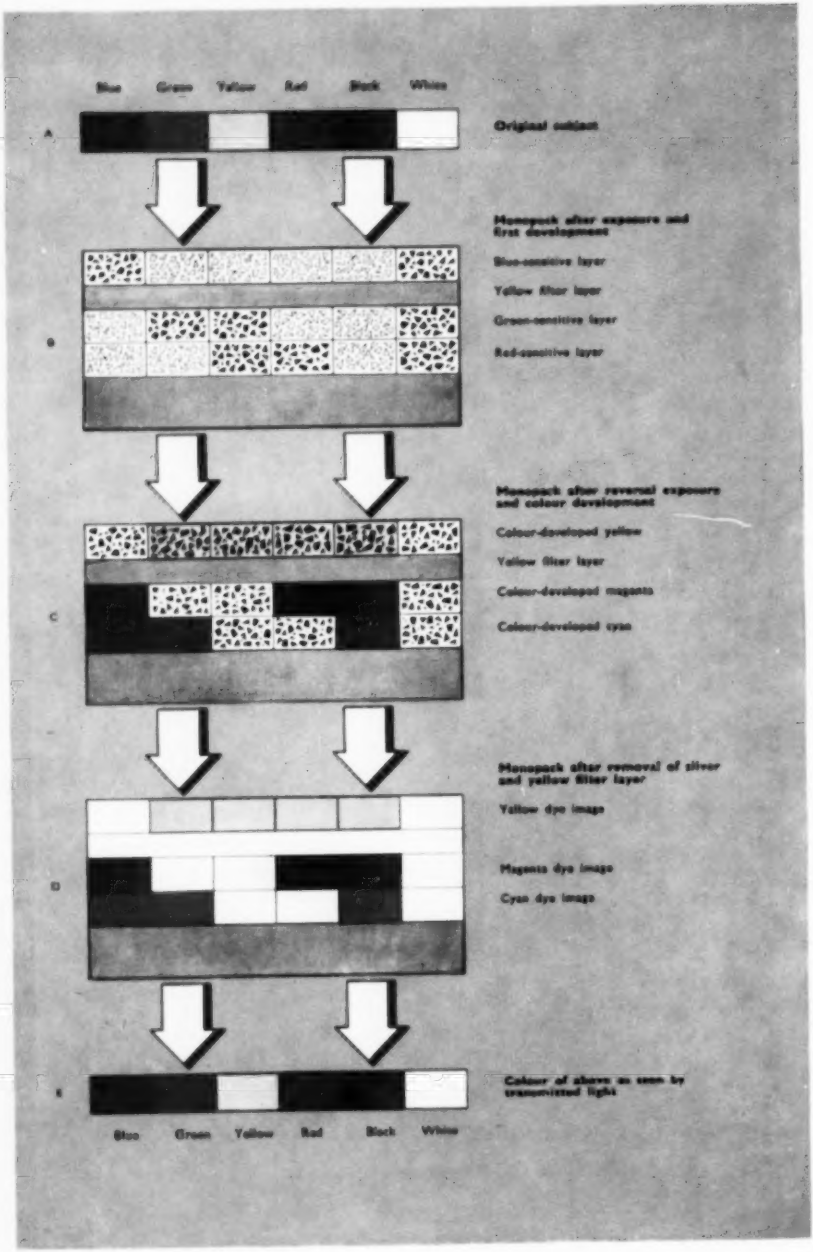


Fig. 6.—Sequence of Operations in Processing Monopack Reversal Colour Film

additive primaries, they can be superposed to give a very great variety of colour sensations, because any pair of them have a common transmission band of colour (Fig. 3B).

A failure to distinguish between these two sets of colours leads to confusion in efforts to understand the principles of colour mixing. The properties of the subtractive colours, like those of the additive ones, have been discovered empirically: it is probably true to say that the idea of the principle of subtractive colours developed with the growth of experience in three-colour printing, and cannot be said to have a single discoverer. In Duhauron's original suggestion for a screen plate, he wrongly proposed the use of red, "yellow", and blue as additive primaries, instead of red, *green*, and blue, but in his 1876 patent [38] he correctly described the need for using the yellow as a subtractive colour along with bluish red and greenish blue for making prints on paper.

B—Basic Principles of Colour Reproduction

In the reproduction of a fully coloured image of an object by photography two processes are involved—

(i) ANALYSIS—The light from the object is divided into its three additive primary components—red, green, and blue—and from each of these an individual negative is produced; these form the so-called *negative separation records*.

(ii) SYNTHESIS—Positives are made from the negative separation records, and are recombined for viewing.

The method of recombination in the synthesis process determines the treatment of the positives and the nature of their colouring, and colour photographs may be resynthesised by what are known as the *additive* and *subtractive* methods.

1. ADDITIVE SYNTHESIS

In additive processes, the silver positive images are retained, and each is viewed through a filter of the same (additive primary) hue as that used in exposing the negative. Viewing must be done additively, i.e. the separate images must be seen in such a manner that light from one does not pass through another. This is shown in the top half of Fig. 7. In this the original picture is recorded through the three taking filters. The resultant negatives are converted to silver positives, and these may then be combined for viewing through filters in three principal ways—

(*a*) By projection of all three simultaneously and in register on a screen (as in Fig. 7) (§ I 4).

(*b*) By presenting them separately to the eye in rapid succession, so that they are combined by persistence of vision (§ I 18(*a*)).

(*c*) By forming a mosaic by dividing the three combinations of positive image and the appropriate filter into a multiplicity of very small areas. These pieces are then arranged over the viewing space as already described (§ I 8), and the whole builds up to a single image.

2. SUBTRACTIVE SYNTHESIS

Stated briefly, the principle of producing colour positives by subtractive synthesis is that the silver in each positive image is *replaced* by a transparent colour, and the three coloured positives thus obtained are superposed in register for viewing. Fig. 7 (bottom half) should be studied in conjunction with Fig. 2 and 3 to understand the principle of this procedure, which is fundamental to all subtractive colour processes. The same principle is also illustrated in Fig. 8A, D. *Each positive layer must be printed in the minus colour corresponding to the additive colour filter used in exposing the negative from which it is printed.* Thus the *blue* filter record must be printed in *yellow*, and so on. Consider e.g. a blue object. This will register only on the negative behind the blue filter and not at all on those behind the other two filters. When the three positives are made, in the appropriate *minus* colours, there is an uncoloured image where the blue object is recorded in the layer printed in yellow, but the magenta and cyan layers below are fully coloured, and through these two together blue light alone passes.

C—Additive Processes

Additive processes may be classified as follows—

(1) Simultaneous projection processes
(2) Successive projection processes
(3) Processes based on optical colour separation
(4) Mosaic screen processes, subdivided into—
  (*a*) Separable screen processes (regular screens)
  (*b*) Integral screen processes, having regular or irregular screens.

In photography itself these processes (except (4)) are little used, but they have great potential importance as the basis of colour television, for which an additive process must be employed.

1. SIMULTANEOUS PROJECTION PROCESSES

These are based on the original idea of Clerk Maxwell and may be employed for still or cinematograph work. They have already been described in §§ I 4, 18(*a*).

2. SUCCESSIVE PROJECTION PROCESSES

These can be used only for cinematography. They have already been described in § I 18(*a*).

3. PROCESSES BASED ON OPTICAL COLOUR SEPARATION

From time to time, processes of colour photography have been demonstrated which depend for their effect on optical refraction, diffraction, or polarisation effects rather than upon image separations by coloured filters*. So far only the lenticular process has passed beyond the stage of being an attractive scientific toy. The others are tedious to operate, and can be viewed only under special conditions; yet they should not be dismissed as for ever impossible of commercial development for large-scale use.

(*a*) *Lippmann's Interference Process*

Strictly, this process (1891) [61] can be classified neither as an additive nor as a subtractive process,

* The results still depend indirectly upon the properties of dyes, because sensitising dyes must be used to make the emulsion panchromatic.

but it is most conveniently included here. The negative is taken on an extremely fine-grained silver emulsion coated on a glass plate, which is floated on a trough of mercury, the emulsion being next to the mercury surface. Light rays incident upon the mirror thus formed and those which are reflected back form stationary waves, and thus they will yield small zones of alternate light and darkness. Latent images will be formed at the points of illumination, one-half wavelength apart, and on development, small silver deposits are produced which act as mirrors, reflecting light of the original wavelength (colour) when the developed transparency is illuminated, preferably against a black background. The best results are obtained by developing lightly, bleaching, and intensifying.

The method is simple and theoretically most attractive. It cannot be widely used because the fine-grain emulsion is very slow, necessitating exposures running to minutes in bright sunlight, and the mercury cannot conveniently be replaced by a solid metal mirror on account of the necessity of ensuring perfect contact over the whole surface of contact of the emulsion and the mirror*. Viewing must take place at a particular angle, and the appearance of the colours may be entirely altered if the gelatin of the emulsion when viewed is not in the same state of swelling, determined by its moisture content, as it was at the moment of exposure. Duplicates cannot be made.

(b) *Lanchester's Microdispersion Process*

A process utilising a diffraction grating was invented by Lanchester in 1895 [51, 92]. The lens L_1 (Fig. 9) throws an image of the distant object upon a diffraction grating G, ruled with parallel opaque lines, some 300 per inch, about twice as wide as the intervening clear spaces. The image upon the grating acts as object for a second lens L_2, which throws its image upon a panchromatic plate S. A narrow (2°) prism P is placed between L_2 and S, and causes each line of the grating G to form a minute spectrum upon S. After exposure, the plate is developed by reversal development, and if it is replaced in the position S and viewed (from the right-hand side in the diagram), the lens L_1 being illuminated by parallel white light, a fully coloured image will be seen.

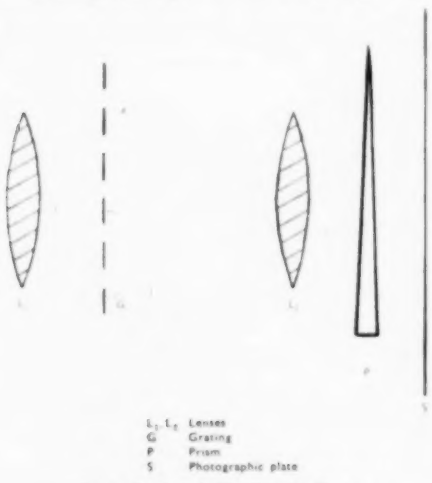


FIG. 9—Lanchester's Microdispersion Process.

* A special sealed dark slide was at one time marketed, into which mercury was introduced to form a backing for the plate, which could thus be used vertically [88]. It is believed that satisfactory results have recently been obtained by the use of aluminised mirrors.

(c) *Wood's Diffraction Process*

This process (1899) [3, 64, 125] also is based on the use of diffraction gratings. *A*, *A*, *A* (Fig. 10) represent

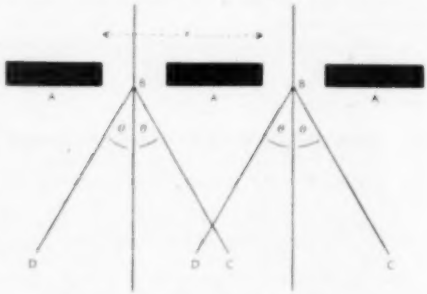


FIG. 10—Principle of Wood's Diffraction Process.

the lines and *B*, *B* the clear spaces of a grating. Light of wavelength λ will be reinforced in a direction BC if the angle θ it makes with the normal to the surface is such that—

$$a \sin \theta = n\lambda$$

(a = width of a combined line and space). Thus for each wavelength in the spectrum there is a particular angle θ at which a beam of that colour can be seen. By suitable choice of the spacing of the lines three gratings can be ruled which will enable any three chosen wavelengths, e.g. red, green, and blue, to be transmitted each at the same angle to the normal. If three such gratings are placed one above the other and in the same vertical plane, when they are illuminated by a beam of white light the three chosen spectral colours will be transmitted on the same vertical line. If the three beams so formed are combined in the eye, white light will result; and if one or other is blacked out, a colour will be seen.

Wood chose gratings with 2000, 2400, and 2750 lines to the inch for the red, green, and blue primaries respectively. Three colour-separation negatives were prepared, and a dichromated gelatin plate was used for the positive. This was printed by placing the plate in contact with the 2000-line grating, and next to it the red-separation negative, the exposure being made through the negative and the grating to parallel light (sunlight). The grating and negative were then removed, and replaced by the 2400-line grating and the green-separation negative, which was then printed. Finally, the blue-separation negative was similarly printed through the third grating. The operation

was completed by washing away the unaffected gelatin in warm water, and then drying. An improved process was to print the third negative on a separate plate, reversed, so that it could be bound up, emulsion to emulsion, in register with the print of the first two. The three grating images, each representing its own colour-separation negative, are thus all present in the same surface, and by viewing in a simple apparatus (Fig. 11) a full colour reproduction is seen. Viewing is thus a three-colour additive process.

A diffraction grating forms an image on either side of the normal, so that in operating Wood's process both eyes can be used if they are placed equidistant from the centre of the lens *L* (Fig. 11).

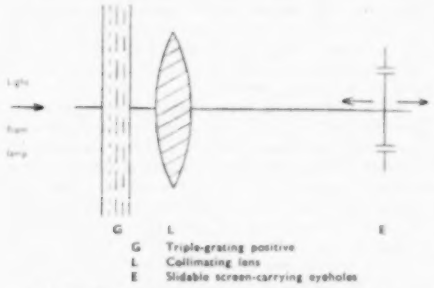


FIG. 11—Viewing Apparatus for Wood's Diffraction Process

The eyeholes *E* in an opaque plate are moved in a direction perpendicular to the illuminated positive *G* until the correct position is found where the red, green, and blue images coincide. The original positive is, of course, colourless and almost invisible when viewed under ordinary conditions.

(d) *Berthon's Lenticular Process*

This process (1909)[12] is the only one employing refraction or diffraction effects which has ever been in general use. It was employed for some years in substandard cinematography (see § I 18(*b*)) and for 35-mm. still pictures in the Leica camera, and has been intensively investigated by several large research organisations, but it seems likely that it will not be employed again in view of the superior virtues of the monopack processes. One side of the film is moulded during manufacture into a parallel series of minute semicylindrical lenses (Fig. 12). It is exposed with this side towards the camera lens, so that each of the minute semicylinders forms a tiny image in the emulsion. The camera lens is covered with a triple filter in which red, green, and blue strips are placed side by side with their edges parallel to the lenticular mouldings on the film. Thus, each of the tiny images in the emulsion will be split into three bands, corresponding to the three filters. For viewing, the film is reversal-developed and projected through a lens having a filter identical with that used for the exposure.

Though the colour reproduction of this process is good, it suffers, like the mosaic screen process, from low light transmission, and an additional disadvantage is the necessity for a specially designed focussing arrangement on the lens to ensure that when it is racked in and out it does not rotate and so throw the filters out of register with the lenticles. This drawback has been eliminated in a recent variation of the process[70], where it is proposed to incorporate certain finely divided polarising crystals in the film base or emulsion. The crystals, e.g. cymophane $BeAl_2O_4$, transmit at least three different spectral colours, as individual polarised beams, according to the particular crystal plane illuminated. Thus the colour separation images are formed in the emulsion without the use of the tripartite filter on the lens.

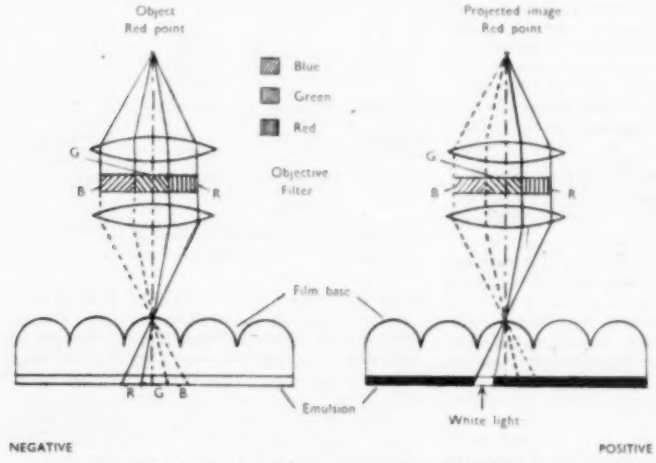


FIG. 12—Principle of Berthon's Lenticular Process. The three-colour filter is focussed on the emulsion by means of the cylindrical lenses impressed in the film

4. MOSAIC SCREEN PROCESSES

The principles of operation have been briefly described earlier (§ I 8), and further details are given below.

Immediately next to the photographic emulsion, on the lens side, is a fine mosaic of minute areas coloured in the red, green, and blue primaries. The emulsion is thus exposed *through* this mosaic, and after normal development to a negative the image is chemically reversed to a positive. On viewing the result as a transparency, after registration with the mosaic,

the image is seen recorded in its original colours (Fig. 5).

(a) Separable Screens

The original Finlay screen process (§ I 12) has recently been reintroduced in this country as the Johnson Colour Screen Process. It has several advantages over the integral screen processes, in which the screen and plate (or film) are inseparable. These include (i) the possibility of producing unlimited duplicates all of the same quality as the original; (ii) a greater latitude in exposure, because the positive is printed from an original negative and not merely formed by reversal; (iii) the possibility of employing different sets of dyes for the taking and viewing screens respectively (the dyes with the best theoretical characteristics for taking are not necessarily those best for viewing, and *vice versa*); (iv) the screen, which is expensive, is needed only for successful pictures, and can be used again if results are unsatisfactory. These characteristics give the process especial value in the preparation of e.g. numbers of demonstration colour slides for medical or scientific work.

(b) Integral Screens

(i) TYPES OF SCREEN.—Many integral mosaic screen processes have been used in past years, and still more have been proposed [13, 65, 131], all differing only in the method of formation of the screen. The screens may be classified under the following six headings—

(α) *Ruled lines.* Joly's original screen plate [74] was prepared in this way.

(β) *Coloured grains dusted on a tacky surface.* The first commercial plate, the Lumières' Autochrome plate (1907), utilised dyed grains of rice starch, while the Agfacolor screen plate used particles of dyed resin (§ I 12). From time to time other types of dyed particle have been advocated, e.g. finely powdered dyed fibres, individual particles of dyes, pollen grains, dead bacteria, and blood corpuscles. However fine the particles may be, the mosaic is usually more apparent than one of regular geometric pattern, because clumps of more than one particle of a single colour are distributed over the surface, according to the laws of probability.

(γ) *Cut sections of bundles of fibres, glass filaments, etc.* Fabric woven from alternately arranged fine coloured filaments may also be included under this heading.

(δ) *Photographically printed lines.* The original Finlay screen, for example, was made by applying collodion, dyeing green, then applying a layer of albumen containing dichromate, exposing to light under a line negative, dyeing (the unexposed portions) red, then repeating the albumen coating, etc., and dyeing blue.

(ε) *Mechanically printed lines.*

(ζ) *Intersecting lines formed by a combination of mechanical printing and dyeing.* The Dufaycolor process is the best example.

The two inescapable drawbacks of the mosaic screen process are its low light transmission and the coarseness of the réseau*. The Dufaycolor film shows a considerable improvement in both these factors over previous screen plates or films. Thus its maximum light transmission is about 25% and it has about 250,000 coloured elements per square inch. Earlier screen plates had maximum transmissions of 7·5–15%†. No other process had such a fine réseau or such a high transmission. The individual starch grains of the Autochrome plate averaged about 0·015 mm. in diameter, compared with an approximate line width of 0·045 mm. in the Dufaycolor réseau, but the "clumping" effect referred to above made the mosaic more visible in the former pattern.

(ii) DYES USED.—The well known types of basic dye, and occasionally some of the triphenylmethane acid dyes, have been employed for colouring the screen elements. Table I shows that there has been little change in the nature of the dyes used over a long period.

Much theoretical investigation has been made into the best spectral characteristics for dyes used in additive processes, and the spectral qualities of screens have been reported[65], but little information is available upon the relation between the theoretical requirements and the properties of available dyes, or upon the synthesis of new dyes with specially designed spectral characteristics. Possibly the range of established textile dyes has been found to supply all that is needed, and certainly the reproduction of colours such as violet, blue, and red in the Dufaycolor films is very good. In the development work preceding the introduction of this film, Baker [10] found that the actual width of the transmission band of each primary was not critical, but it is important that the three bands overlap by about half their width. The use of wide overlapping bands, in conjunction with the entirely contiguous lines of the réseau, which require no interstitial blacking-out, accounts for the high light transmission of this screen (Fig. 13).

TABLE I

Dyes used in Screen Mosaic Elements

Plate or Film	Red	Green	Blue
Joly (1894) [74]	Chrysoidine (*C.I.* 20)‡	Ethyl Green (*C.I.* 685) Chrysoidine	Water Blue (*C.I.* 707)
Omnicolore (1907)	Rhodamine B Auramine	Malachite Green (*C.I.* 657) Auramine (*C.I.* 655)	Methylene Blue (*C.I.* 922)
Dioptichrome (1908) [9]	Rhodamine B (*C.I.* 749)	Malachite Green	(?)
Dufaycolor (1928) [22]	Basic Red N extra (Kuhlmann)	Malachite Green Auramine	Malachite Green Crystal Violet (*C.I.* 681)
I.G. screen (1935) [70]	Phenosafranine (*C.I.* 840) Auramine	Methylene Blue Auramine	Methylene Blue Crystal Violet

‡ The use of this orange dye may be accounted for by the difficulty of obtaining sufficient sensitivity to red at that date.

† Surmised identity of dyes, based on colour reactions given by Wall [158].

* Each primary ray must pass through a filter absorbing at least 66⅔% of the total light. In practice, this is less noticeable than might be expected, because of the adaptability of the eye to changes in brightness. Neither is the Dufaycolor screen noticeable at normal viewing distances.

† The Dufay Dioptichrome plate of 1908 was said to have a 20% transmission value (Mees and Pledge [107]).

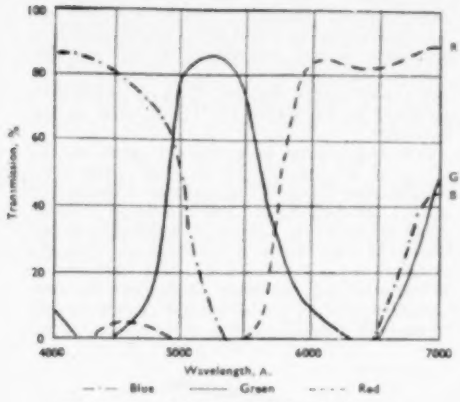


FIG. 13—Transmission Curves of Dufaycolor Primaries

(iii) MANUFACTURE OF DUFAYCOLOR FILM—The manufacture of the Dufaycolor film [10, 26–28, 33–37, 121, 129, 130] is carried out by continuous processes involving the following stages (Fig. 6)—

(α) Coating of cellulose acetate film base, in continuous 1000-ft. lengths and 21 or 42 in. wide, with a layer of collodion, dyed blue.

(β) Printing, diagonally across the collodion surface, of fine parallel lines (20 or 23 per mm. according to type of film) in a greasy ink, by passage between an etched steel and a rubber roller.

(γ) Drying of the ink for 1 hr. under carefully controlled conditions.

(δ) Removal of the dye in the spaces not covered by the ink lines by passage through a bath of methyl alcoholic potassium hydroxide, or of a mixture of this with aqueous hydrogen peroxide [34].

(ϵ) Redyeing of the decolourised spaces in green.

(ζ) Removal of ink by a solvent.

(η) Printing of a second set of parallel lines in greasy ink at right-angles to the first set, the lines being broader and the intervening spaces narrower than in the first set, to ensure correct colour balance.

(θ) Drying of ink lines.

(ι) Decolourising as in (δ).

(κ) Dyeing of decolourised spaces red.

(λ) Removal of second set of ink lines.

(μ) Varnishing of surface to prevent diffusion of dyes into the emulsion and to effect slight optical diffusion of the image of the réseau on the emulsion.

(ν) Coating, on the surface of the varnish, with a panchromatic emulsion, rich in silver (to give dense blacks).

(iv) DUPLICATION OF DUFAYCOLOR FILMS—For professional cinematograph work, improvements were made which enabled duplicates to be produced, so that the original film could be processed to a negative and an unlimited number of positive copies taken from it. Previously it had proved impossible to make satisfactory duplicates of mosaic films by contact printing because of (α) the moiré effects which appear when two réseaux are placed in contact, and (β) a slight diffusion of the silver image beyond the individual elements under which it should occur.

The formation of moiré patterns was overcome in a simple but elegant manner by a method devised by Harrison [83]. By control of the size, shape, and intensity of the illuminant, and the distance apart of the original and the copy film, the individual elements of the réseau on the negative may be made just to blend together by optical diffusion at the surface of the réseau on the copy film. The spread of the silver image was prevented by a developing process, devised by Spencer, in which development commenced at the inner surface of the emulsion nearest the réseau.

A mixture of red, green, and blue monochromatic light is employed for printing to ensure that the light transmitted by each element of the réseau in the negative affects only an element of the same colour in the positive. On account of the wide wavebands transmitted by the dyes employed, if ordinary light were used, each element of the negative would tend to print on to parts of the positive emulsion behind elements of the other two colours. This would cause spreading of the image and desaturation of the colours. The monochromatic light source consists of a didymium filter behind which are arranged a high-pressure mercury-vapour lamp with an opal glass screen and a 500-w. tungsten lamp behind a red filter.

D—Subtractive Processes

1. SEPARATION NEGATIVE PROCESSES

These processes work according to the principles outlined in § II B 2. Prior to the recent introduction of monopack material, colour prints on paper or film could be made only by transferring three transparent colour separation images into superposition. Processes worked on this principle, e.g. Carbro, dye transfer, Technicolor, are of considerable commercial importance, and are capable of giving excellent results. Three operations are involved—formation of an image in gelatin, coloration of the image, and transfer to a permanent support. Details of these are given later in this paper (§ III 6).

The negatives are exposed through the three additive primary filters, either simultaneously (in a one-shot camera) or in succession. For much commercial colour-blockmaking work, the original may itself be a colour photograph, i.e. a positive monopack transparency.

2. MONOPACK PROCESSES

These have already been considered in part (§§ I 15 and II D 2). A typical monopack material (Fig. 8B) consists of a base on which there are coated, in order outwards, a red-sensitive layer, a green-sensitive layer, a yellow filter layer, and a blue-sensitive layer. This arrangement is common to the two classes of monopack material—(a) reversal and (b) negative-positive films.

(*a*) *Reversal Films*

The sequence of operations in producing the final colour positive is shown in Fig. 8. In all present-day films the final images are in the form of dyes produced by a colour development process, as described in more detail in § III 5, and there are two general methods for its operation, the colour-producing reagents being either (i) used in the developing baths or (ii) incorporated in the emulsion itself during manufacture.

(i) COLOUR COUPLERS IN DEVELOPING BATHS—This method avoids the difficulties involved in adding these materials to the emulsions and enables greater control to be exercised during processing; it is employed for the present Kodachrome and Ilford Colour films. After exposure the whole film is developed in the normal manner for black-and-white emulsions, and then each layer of residual silver halide is separately re-exposed to light and colour-developed, as described in § III 5 (*e*).

(ii) COLOUR COUPLERS IN THE EMULSIONS—By this method the processing is very much simplified. The Agfacolor film uses colour couplers made substantive to the emulsion gelatin by the presence of certain "anchoring" groups in the molecule. Kodak's Ektachrome and Kodacolor films use colour couplers dispersed in an organic compound which is insoluble in, but permeable to, water, e.g. tritolyl (tricresyl) phosphate (§ III 5).

(*b*) *Negative–Positive Processes*

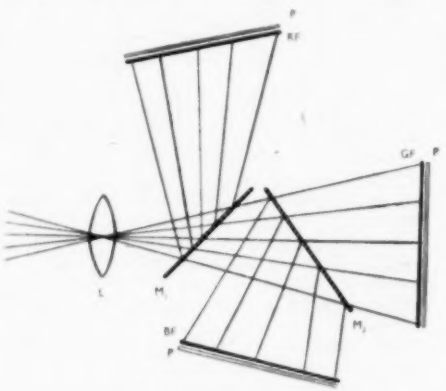
These, usually referred to as "neg.–pos." processes, were introduced later than the reversal processes on account of difficulties in overcoming the colour degradation which occurs at the printing stage (see § II F 6). Typical films are the Agfacolor (substantive couplers) and Kodak's Ektacolor and Kodacolor (dispersed couplers). The sequence of operations in processing a typical neg.–pos. film is shown in Fig. 4. Agfacolor and Kodacolor films provide colour prints on paper. The last-named formerly used contrast-reducing silver masks, but now both Kodacolor and Ektacolor use coloured couplers for automatic masking (§ II F 6(*b*)).

E—Colour Cameras

1. THE STILL CAMERA

Three individual colour-separation negatives are essential where colour positives are to be made by a photographic transfer process or by printing from half-tone blocks*. These must be made with a normal plate camera, preferably with some form of sliding back, in which the plate-holders can be moved easily into position in succession. For separation records of a moving object, an exposure may be made on one of the materials giving a direct colour transparency, and this may then be copied on to the separation negatives. This procedure obviously increases the inevitable departures from faithfulness to the colours of the original, and so the use of a one-shot colour camera may be preferred. In this, all three plates are exposed simultaneously through a single lens and the appropriate filters. With steady improvements in monopack colour transparencies the one-shot camera may eventually become obsolete, but this is not so yet†, and a number of designs† have been produced in Britain, in the U.S.A., and on the Continent.

One-shot cameras have a history nearly as long as colour photography itself, since Duhauron's original suggestions (§ I 7) included a description of one. In spite of the work of a legion of subsequent inventors in proposing modified constructions, the present-day instruments are basically similar in design to Duhauron's. Fig. 14 illustrates the principle of operation, which depends upon separation of the light beams by a system of semi-transparent ("half-silvered") mirrors.



L	Lens
M_1, M_2	Semi-transparent mirrors
P	Plates or films
BF	Blue filter
GF	Green filter
RF	Red filter

FIG. 14.—Diagram of One-shot Colour Camera

To avoid parallax errors there must be only one lens, and the separation of the light into three beams must occur behind it. Much ingenuity has been exercised in efforts to reduce the inevitable bulkiness‡ of the instrument, and precision standards of workmanship are essential to ensure that the three images are exactly the same size when printed. Some of the principal difficulties to be overcome in the design of these instruments are—

(*a*) Formation of double images by additional reflection from the back surface of the half-silvered mirrors. This has been overcome either by coating this surface with a gelatin filter layer coloured with the complementary colour to that to be reflected,

* Description of the preparation of separation negatives or of three-colour blocks is outside the scope of the present paper, and must be sought in the standard textbooks or in the literature issued by the manufacturers of photographic materials. Schneider[124] describes the preparation of separation negatives and blocks from Agfacolor transparencies.

* Manufacture in the U.S.A., indeed, commenced only in 1935, and reached a peak production level in 1938[39].

† For illustrations and descriptions of commercial instruments, see e.g. Coote[38] and Spencer[122]. The very extensive patent literature on the subject is surveyed by Wall[123] and Friedman[51].

‡ A more compact and symmetrical design can be achieved by the use of mirrors arranged in X-form, but though this principle has been described it does not seem to have been applied much in practice.

or by using, in place of glass, thin collodion films (pellicles), kept taut by springs in a metal frame [53].

(*b*) Uneven illumination of the plate, due to the difference in the angle of incidence of rays at the two ends of a mirror which is inclined at an angle to the axis of the lens. This effect must be minimised by grading the density of the semi-reflecting metal layer.

(*c*) Differences in equivalent lengths of light path of the three beams, since they do not all traverse the same thickness of glass. This leads to slight variations in the focal distances of the image, which must be allowed for in the placing of the plate-holders.

Gold is said to be the best metal for forming the semi-reflecting mirrors, because it transmits green light and reflects red and blue. The second one may be of silver, which transmits blue and reflects red, or of aluminium*. Panchromatic plates may be used behind red, green, and blue filters, or a panchromatic plate behind red and orthochromatic plates (i.e. blue-, green-, and yellow-sensitive) behind yellow and magenta filters, for the red, blue, and green records respectively. The latter arrangement is said to give better results [39].

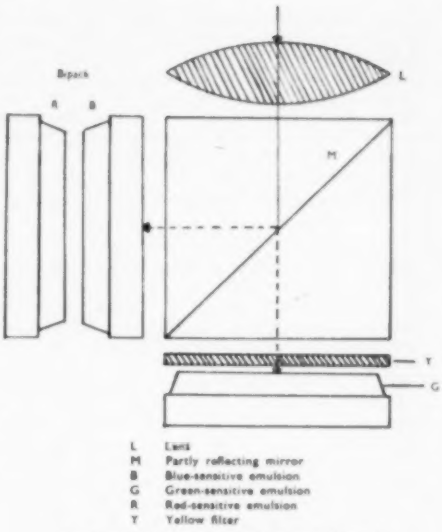


FIG. 15.—Principle of Technicolor Bipack Camera*

2. THE TECHNICOLOR CAMERA FOR MOTION PICTURES

The possibilities of variation in design when mirrors are used are comparatively limited, but a truly bewildering variety of beam-splitting prismatic devices, often of great complexity, may be found in the patent files. Some of these have been employed in additive colour cinematography, but at the present day only one design appears to have survived the test of practical use. This is a simple arrangement of two 45° prisms, used in the Technicolor motion picture camera. Prisms are impracticable in large plate cameras because of their weight, but they can be used in 35-mm. film cameras, and their rigidity and permanence offer an advantage over mirrors or pellicles.

The Technicolor camera [87, 93, 124, 137, 140, 141] is a remarkable and somewhat complex instrument with an optical system specially computed by the British firm of Taylor, Taylor, Hobson & Cooke to give superior definition to that normally required by a black-and-white film, to compensate for the loss of definition inevitable in the colour printing process (Fig. 15).

The prism and lens system is designed so that the whole of the green waveband is brought to the same critical focus, because this image determines the visual sharpness of the final picture. The red and blue wavebands, which are reflected by the gold semi-reflecting mirror, are then brought to a focus in one plane and recorded on the bipack film. The filter between the emulsions of the bipack takes the form of a red dye layer on the surface of the blue-sensitive film. A magenta gelatin filter may in addition be placed in front of the bipack. The directly transmitted green light reaches the third emulsion through a green filter. An alternative arrangement has, in place of the green-sensitive film and green filter, a bipack behind a yellow filter. The first emulsion is a thin panchromatic one, on top of which is a green dye layer (as a filter), and then the green-sensitive emulsion. The thin emulsion records a weak red-green record; this may be used to print a weak black positive in silver, which is combined with the final print as a so-called "key" record to give better blacks and deep shadows. The films are specially made by Eastman Kodak, and the red and green records are taken on emulsions of the same type.

The Technicolor camera is an apparatus of high precision, though it is a very bulky and heavy piece of equipment. The mechanism is noisy, and the camera must be shielded by a soundproof box. The whole assembly, which weighs about 300 lb., needs a specially designed tripod and a small crane for moving the camera. Constant attention by a skilled technician is essential to ensure its efficient operation.

The printing of Technicolor films is described briefly in §III 6 (*c*) iii.

F—Colorimetric Principles

Much attention has been given in recent years to the application of quantitative colorimetry to colour photography [e.g. 25, 57a, 60, 77, 96, 97, 137, 161]. The subject is abstruse, and only a broad outline of the general principles involved can be included here†. Understanding of the chromaticity diagram is necessary, and for the benefit of those who may not be familiar with its significance, a short account is given below.

* See e.g. Yarwood [169], who examined various metals, sputtered or evaporated on to glass in vacuo.

* Fig. 10 in *FIAT* 976 (p. 97) [138] shows an earlier arrangement.

† For a good summary, see Wright [169]. For an earlier treatment of this subject, see e.g. Mees and Pledge [101]. An interesting account in simple terms is given by Chamberlin [20].

Any colour can be exactly defined by means of the familiar type of spectrophotometric curve showing relative intensity plotted against wavelength (e.g. Fig. 10), but for many purposes other types of specification are preferable. The two which are important in the present discussion are the trichromatic and monochromatic systems. Both start with the basic assumption that colour is three-dimensional and therefore any colour can be uniquely specified by three independent variables.

1. THE TRICHROMATIC SYSTEM

A colour can be matched by a mixture of three primary colours. The choice of primaries is wide, the only limitation being that no two must produce the third when they are mixed. To define a colour, then, it is matched in an instrument called a *colorimeter*, against measured proportions of three chosen primaries. The figures thus obtained are subjective, and they must be transformed by arithmetical treatment into terms which are a permanent objective record of the original colour. First of all the results which would be obtained by a statistically "standard" observer must be calculated. Secondly, these results must be expressed in terms of an internationally agreed set of standard primary stimuli, known as the C.I.E.* X, Y, and Z primaries. The final set of figures are then known as the X, Y, Z tristimulus values for the colour under test. The X, Y, and Z primaries are imaginary. They are purely mathematical concepts, representing no colours within human experience, and are so chosen that only positive quantities are needed for matching all colours; i.e. they lie outside the spectrum locus (Fig. 16, 21). Real primaries require to be used in negative amounts for matching some colours, i.e. to be added to the colour being matched. This is necessary when the test colour, though of the same hue, is more saturated than the nearest mixture of the three primaries.

2. MONOCHROMATIC SYSTEM

This specifies a colour by the three terms—(*a*) *dominant wavelength* (hue), which is the wavelength of monochromatic spectrum light which, when mixed with white, will exactly match the test colour†; (*b*) *purity* (saturation or "vividness"), which is defined by the proportion of monochromatic spectrum light in admixture with white in the matching colour; and (*c*) *luminance factor* (lightness), which is defined as the total amount of light compared with that reflected by a magnesium oxide screen under the same illumination.

Such a method of specification corresponds more with the normal visual evaluation of colour than does the trichromatic specification, and thus gives a set of figures whose meaning can be more readily visualised than the related set of tristimulus values.

3. THE CHROMATICITY DIAGRAM

When it is desired to specify the "colour quality" or *chromaticity* of a colour, the tristimulus values X, Y, Z are replaced by $x = X/(X + Y + Z)$, etc. Since $x + y + z = 1$, any two of these are sufficient to specify the chromaticity*. The two chosen values may then be plotted on rectangular coordinates. The spectrum colours form a well defined curve, the *spectrum locus* (*AHEGDFB*, Fig. 16), when plotted in this way on what is known as the *chromaticity diagram*.

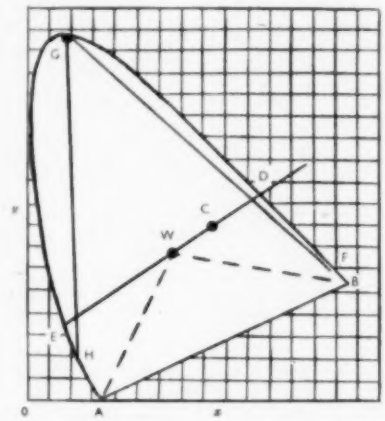


FIG. 16.—Chromaticity Diagram showing the Spectrum Locus, and its Application to Colour Mixtures

Colours along the straight line AB, formed by mixing spectral red and blue in different proportions, are known as *purples*. They are not found in the spectrum, and are defined by their *complementary* wavelength, found as described below (*d*).

Such a diagram is not only a useful pictorial representation of colours, but it has valuable quantitative properties. Those with which we are immediately concerned may be summarised as follows—

(*a*) The dominant wavelength of any colour whose chromaticity point is C is that of the point D at which the line WC, when produced, cuts the spectrum locus, W being the chromaticity point of white.

(*b*) The purity ("excitation purity") of the colour C is given by the ratio CW/DW.

(*c*) Mixtures of any two colours are all represented on the straight line joining the points for those two colours. Thus all colours obtained by mixing D with white lie on the line DW, and all colours formed by mixing F and G lie on the line FG. By the same reasoning, all the non-spectral purples are found inside the triangle WAB.

(*d*) The complement of any colour is found by producing the line joining that colour with white until it cuts the spectrum locus on the other side. Thus the complementary to any colour on the line WD is represented by a point on the line WE.

(*e*) The above arguments may be extended to include mixtures of any number of different colours. Thus all colours whose formation is

* Commission internationale de l'Éclairage. In the U.S.A., the abbreviation I.C.I. (International Commission on Illumination) has been used until recently.

† All visible colours, except purples (see § II 7 5), are thus impure examples of one hue of the spectrum.

* Thus luminance is separated out as the third variable and is not considered.

possible by mixing three individual colours lie inside the triangle formed by joining the respective points, e.g. *FGH*. It is at once clear that *no* set of three primaries can match all the spectral colours. The exact physiological reason for this is still a matter of debate, though it is undoubtedly related in some way to the nature and number of the response curves of the retinal receptor mechanism.

4. COLOUR REPRODUCTION BY COLOUR PHOTOGRAPHY

(*a*) *Additive Process*

We are now in a position to study the use of the chromaticity diagram in colour photography. The simplest case to consider is an additive process with separation negatives and positives, employing in place of colour filters three monochromatic spectral radiations, say *F*, *G*, *H* (Fig. 16), for the synthesis process. The problem resolves itself, firstly, into the most suitable choice of these primaries. The triangle *FGH* should be as large as possible, provided that *F* and *H* are not too near the ends of the locus, because colours there are too dark. The line *FG* should, moreover, lie as close to the locus as possible, because saturated yellows and oranges are common in Nature, whereas saturated blue-greens are not; the closeness of *GH* to the locus is therefore less important.

The choice having been made, it is possible to calculate data for curves showing the relative proportions in which the three chosen primaries must be mixed to give a match with any wavelength of the spectrum. Such "mixture" curves for the primaries *F*, *G*, *H* are shown in Fig. 17*. Details of the methods of calculation must be sought in textbooks on colorimetry.

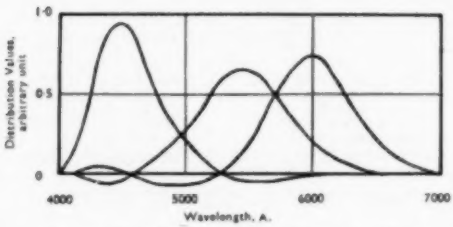


FIG. 17—Mixture Curves for Additive Primary Transmissions

These curves may be regarded as showing the extent to which each one of the set of three radiations of given dominant wavelengths should be controlled in intensity to produce the desired result. It is then clear that they also represent the curves of emulsion sensitivity† required for the three separation negatives if the analysis into three records is made by using plates of different sensitivities, and no filters are used in the camera. This is because the extent of light action on a negative emulsion ultimately controls the amount of synthesis primary allowed to pass, assuming that the three negatives and the three positives are all developed to a contrast represented by a γ-value of unity. (For information on the control of contrast, see a textbook on photography, e.g. Mees [105].)

Actually, of course, the same emulsion is usually employed for all three records, and the equivalent of differential emulsion sensitivities is obtained by employing three filters. It is then necessary to determine the sensitivity curve for the emulsion right across the spectrum, and hence to calculate the transmission curves of the filters necessary to produce the theoretical resultant sensitivities. In the integral mosaic screen process, where the same filters are used for analysis and synthesis, the process should be to choose filters of dominant wavelengths *F*, *G*, *H* and then to adjust the emulsion sensitivity to suit the three filters. Further, for viewing, colour filters are used in conjunction with white light and not monochromatic radiations. The argument, however, is the same, except that the points *F*, *G*, *H* will lie inside, and not on, the spectrum locus, because filter primaries are less saturated than spectrum light of the same hue.

The control of both reproduction primaries and emulsion sensitivity is in practice restricted by the properties of the filter and sensitising dyes actually available, and wide departure from the theoretical ideals may therefore be necessary.

(*b*) *Subtractive Process*

As already stated, it is theoretically impossible for any three-colour process to record *all* colours accurately, and the imperfections of the available dyes still further remove the possibilities of perfection. Thus the ideal subtractive-synthesis dyes would have transmission curves with sharp cut-offs like those of Fig. 18, so that each pair together would transmit one-third of the spectrum. In fact, actual dyes are far different from this ideal (e.g. Fig. 19).

The description "subtractive", applied to the minus primary colour filters, refers only to their action on light outside the eye. The visual appearance of a *minus* primary colour is the result of *additive* action inside the human optical system. Thus, the minus green primary is seen as magenta because both red and blue are transmitted simultaneously by it to the eye. We can thus still apply the same theoretical treatment as that for the additive photographic process, bearing in mind that the three reproduction primaries used are those obtained by combining the three minus dyes in pairs. The primaries found in this way for the Agfacolor film are shown in Fig. 20. The position of such primaries in the chromaticity chart will then determine the range of colours theoretically realisable in the positive, in the manner already described for the additive process.

The actual changes which do take place in the appearance of colours by reproduction with the subtractive process have been extensively studied in the case of Agfacolor film, and the results may be found, together with a discussion of the theoretical aspects of colour reproduction, in a

* The curves of Fig. 17 are for a spectrum with equal energy distribution throughout.
The negative portions of these curves are an expression of the fact, already noted, that some very saturated colours cannot be matched without first being mixed with a desaturating colour.

† Photographic emulsions cannot be negatively sensitised, so that the negative portions of the curves must be ignored. The result is that most saturated colours, especially blue-greens, will be desaturated in the final record.

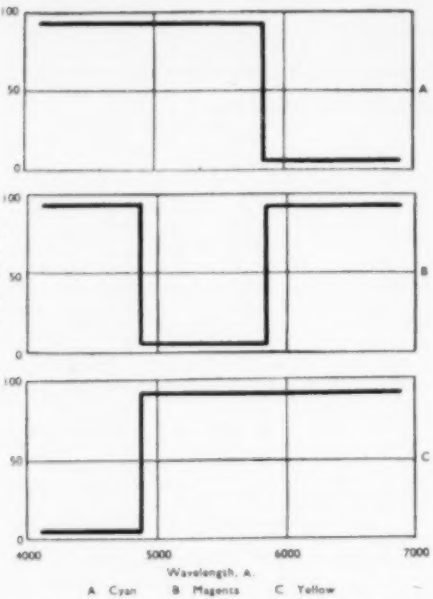


FIG. 18.—Absorption Curves of Ideal Subtractive Primaries

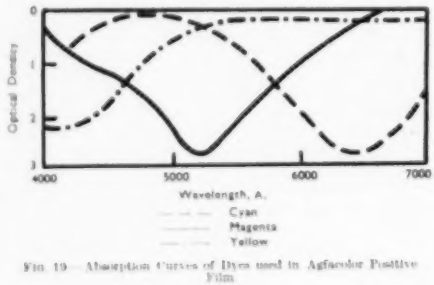


FIG. 19.—Absorption Curves of Dyes used in Agfacolor Positive Film

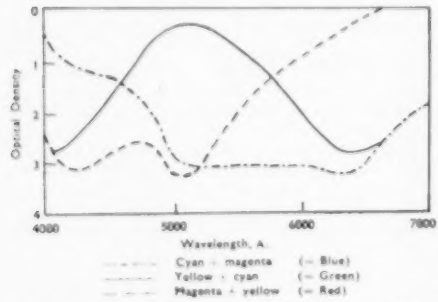


FIG. 20.—Primaries obtained by combining the Agfacolor Positive Dyes in Pairs

recent report [77]. In general, there is a loss of saturation of all colours in reproduction, and sometimes a change in hue; e.g. violets are reddened in reproduction. This would be expected because of the unwanted red transmission of the minus red dye.

In Fig. 21 is shown the approximate area (shaded) of the chromaticity chart that is reproduced by a typical colour film; the chromaticity points of several common pigments of various hues, representative of many of the colours met with in objects of everyday experience, are superimposed to show that such a film is capable of reproducing these colours faithfully.

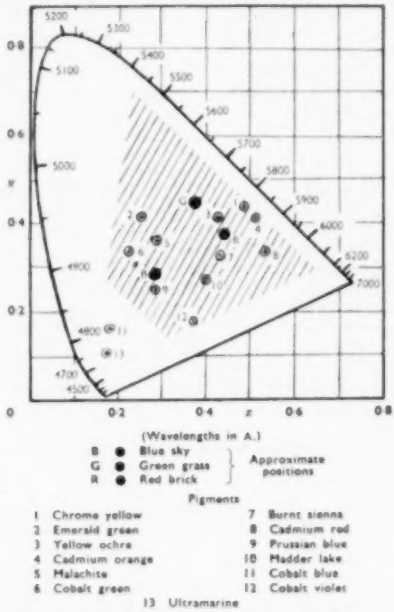


FIG. 21.—Chromaticity Chart, showing Positions of Common Colours and Approximate Range of Agfacolor Films (shaded area)

Very similar results have been obtained by Clarkson and Vickerstaff [25] in an interesting investigation of the theoretical and actual colour-reproducing possibilities of the Kodachrome and Technicolor processes. Using fabrics dyed with a range of the brightest dyes, they found that many of these lay outside the reproduction range of Kodachrome film, especially at high luminance factors. Reds, magentas, and greens became yellower in reproduction, while blues became greener. Deep blues became greyer (i.e. less saturated). There was a loss in purity in all cases, especially with blues. The Technicolor process was found to be able to reproduce a much wider range of colours, covering all the most important regions. The reproductions with both processes lay inside the values calculated as theoretically possible from

the spectral characteristics of the dyes in the films. Proceeding from this observation, these authors then examined the absorption curves of a number of available dyes, both experimental samples and regular commercial types, and were able to select three which, by their satisfactory spectral characteristics, would, if they could be used in a subtractive colour-photographic process, give satisfactory reproduction of nearly all the colours met with in common experience. The selected dyes were chosen without regard to their suitability in other respects, but these observations give promise that eventually the synthesis of improved dyes will enable adequate colour reproduction by photography to become possible.

The theoretical colorimetric requirements are a useful guide to the organic chemist in his attempts to synthesise dyes for colour photography, but the choice of the photographic chemist who uses them is circumscribed by many exacting physical and chemical specifications besides the requirements of ideal hue. Even if dyes with hues of exactly theoretical specification could be used, their suitability might still be modified by more or less unmeasurable influences, partly psychological in nature, which enter into the judgment of the appearance of a picture. Whatever the scientist may say of the truthfulness of a reproduction to the original, it may be judged good or bad by factors outside his calculation. The human visual mechanism has great powers of accommodation to the prevailing illumination, which the photographic system has not. Thus a colour photograph, showing e.g. flesh tones or a light-coloured garment, taken in sunlight on grass may show an overall greenish tint, which, though a truthful physical record of the scene, would be rejected as unpleasant by the eye. Less serious, but still noticeable, are the effects produced in the colour record by variation in the colour temperature (§ II 5) of the daylight or artificial illumination, because films are sensitised for a given colour temperature only. Filters can be used for correction of unsuitable light, but to cover every possible variation in this way is impracticable. Even in daylight the colour temperature may vary considerably according to the cloudiness of the sky; the eye may not be aware of the changes, but the camera records them.

Nevertheless, the results obtained by modern processes are undoubtedly pleasing to the eye, in spite of their theoretical faults*. Two reasons may be given for this—the reproduction is seldom compared directly with the original; and most colours seen in normal life are unsaturated, many indeed lying well within the range theoretically reproducible. (Spectral colours or coloured lights of high saturation rarely have to be recorded.) Many of the commonly used artists' colours, for example, have low saturation (Fig. 21), yet we are not normally conscious of any lack of truthfulness from this cause in pictures painted with such pigments.

* One observer reports [1] that the Agfacolor cinematograph film (produced by the negative–positive process) showed outstandingly good "rendering of green landscapes and skies with clouds. Flesh tints tended to be heavy and somewhat variable in the outdoor scenes, but frequently more natural in the interiors. It would appear that the range of the process is still inadequate for entirely pleasing rendering of outdoor subjects in general."

5. COLOUR TEMPERATURE

The colour temperature of a source of light is defined as the temperature in degrees absolute (°K. = °C. + 273) at which a black body would radiate light to give a visual match. Colour films are normally sensitised for either sunlight or artificial (Photoflood) light. Some typical colour temperatures are given in Table II, from which it will be apparent that a "daylight" film may give distorted colour values under e.g. overcast conditions. Contrary to popular belief, the general illumination is *bluer* just after sunrise or just before sunset than during the rest of the day, because of increased scattering. Only the *direct* rays of the sun are redder at those times [113].

TABLE II

Colour Temperatures

Source	Colour Temperature (°K.)
Tungsten lamp (100 w.)	2835
Tungsten lamp (500-w. projection type) ...	3190
Tungsten lamp (Photoflood type) ...	3350–3450
Flashlight bulb	3500–4000
"Integrated" daylight from surrounding objects on the ground [113]	ca. 4800
White-flame carbon arc	5000
Average noon sunlight	5400
Light from totally overcast sky	6800
Light from clear blue sky	12,000–27,000

6. DUPLICATION OF COLOUR PHOTOGRAPHS

When duplicates are required from additive processes (except, of course, where the mosaic screen is an integral part of the plate or film) or from e.g. Technicolor negatives, no difficulties arise, but where they must be taken from an already coloured record, serious colour distortion may occur: the errors in the colour record due to the imperfections of hue of the dyes will be increased when duplicates are made. We may now consider the steps which may be taken to minimise the consequences of these imperfections. Two types of monopack process are concerned, negative–positive and reversal, and also subtractive printing from separation negatives.

(a) *False Hues in Negative Monopacks*

In a negative–positive monopack process, the dyes used in the *positive* do not have any part in the actual duplication process, and their choice is therefore independent of it. The operative factors are the *hues* of the dyes used in the *negative* and the *sensitivities* of the *positive* emulsion layers. To eliminate the magnification of colour errors caused by faults in the dye absorptions it is clearly necessary to ensure that the dye in each negative layer controls the amount of dye later formed in the corresponding positive layer only; it should have no influence at all on the other two layers. Ideally this can be ensured by having dyes in the negative with no overlapping absorption bands, and using narrow sensitising ranges for the positive layers, each covering only the maximum absorption of the dye in the corresponding negative layer (Fig. 22).

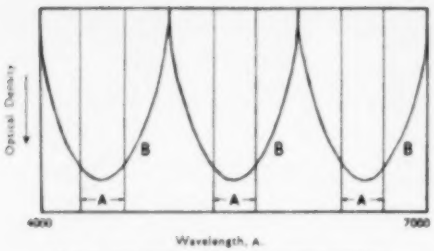


FIG. 23—Ideal Dyes for Negative Monopack and Sensitivity Ranges for Positive Layers

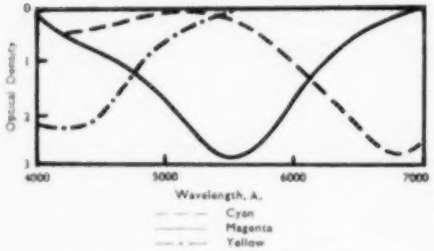


FIG. 23—Absorption Curves of Dyes in Agfacolor Negative Emulsions

Owing to the difficulty of sensitising only to a narrow spectral band, it may, however, be necessary to have the absorption maxima of the negative dyes spaced more widely apart than those normally used for viewing. This can be done without regard to the effect on the hue, because the negative need not fulfil any criteria of visual colour truthfulness. The Agfa laboratories found [126] that dyes with the absorptions shown in Fig. 23 gave good results in actual practice. Wider separations were not practicable for other (chemical) reasons.

(*b*) *Masking*

When preparing a duplicate positive direct from an original positive transparency by the reversal process, the device of using false hues in the negative cannot, of course, be employed, and degradation of colour values is inevitable. Even a casual glance at a duplicate colour transparency prepared thus, alongside its original, shows that many colours, especially bright blues and greens, have become duller, and that the yellows, oranges, and reds appear brownish. The reason is attributable to the absorption spectra of the minus primary dyes used. These are never ideal, and always absorb some of the light which should be freely transmitted. The cyan dye, especially, usually shows appreciable absorption of blue and green (Fig. 24), and magentas absorb blue and red; this is largely responsible for degradation of colour values in duplication. The yellow dye is usually more satisfactory, absorbing little or no red and green.

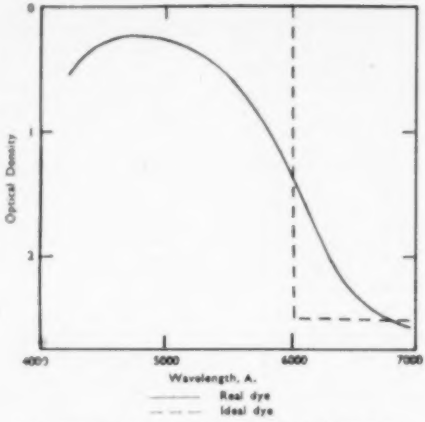


FIG. 24—Light Absorption Curve of a Typical Cyan Dye

A red image would absorb blue and green light; therefore the cyan image, absorbing as it does some of these two colours also, is equivalent to a true cyan image plus an exact, but weaker, duplicate in red. Similarly, a magenta image has an equivalent or "shadowing" image in green. These undesired "shadow" images will record in the appropriately sensitised layers when the positive is duplicated by reprinting, and the result will be a degradation of hue, which will become worse with each successive duplication. In practice, for this reason only a first duplication of positive transparencies is practicable.

Correction of this fault, failing improvement in the dyes themselves, which has not so far been achieved, can be partly achieved by *masking**, a device first suggested in connection with three-colour printing by Albert as far back as 1899.

The problem may be studied in connection with printing, by white light, from a negative monopack transparency with cyan, magenta, and yellow layers. Considering the cyan record of the original, the aim must be to eliminate or "neutralise" the red "shadow" image of the true cyan positive. This can be done by preparing a *negative* of it, also in red and of similar contrast range, and binding this up in register with the original. If the contrast of the negative is correctly controlled it will exactly "neutralise" the red positive, and the net effect will be a uniform red coloration covering the whole picture. This coloration can be corrected by a suitable alteration (increase in blue–green) in the colour of the printing light. By a similar procedure the unwanted green transmission of the magenta record can be compensated for.

An elegant method of carrying out such masking techniques automatically in printing from a colour

* Considerable research has been carried out upon this process in recent years. For a full discussion of the mathematical theory and the practical details, etc. see e.g. Spencer [128] or the patent literature e.g. 86–87, [88]. Good surveys of the whole subject are given by Miller [129] and by Robinson [130].

negative has been described recently by the Eastman Kodak research laboratories [57]. This utilises colour couplers, incorporated in the emulsion during manufacture, which are themselves coloured to the required hue of the mask, but when they are acted upon by the colour-forming developer the original colour is destroyed and the required minus primary colour formed instead. Thus the coupler which forms the cyan dye is itself coloured orange–red, and that which forms the magenta is yellow*. The chemistry of these compounds will be described in § III 5 (*d*). Another chemical device for automatic masking is illustrated in a proposal [67] to counteract the excessive orange tint (i.e. blue absorption) of the magenta dye by giving a very low *red* sensitivity to the blue-sensitive layer. Thus red objects will record faintly in this layer, and so reduce the amount of the yellow dye produced therein.

These are the most recent examples of the masking technique. A somewhat more elaborate variation of it has been known for nearly twenty years, for correcting the hue of the subtractive colours used in printing by one of the transfer processes from separation black-and-white negatives. Thus if it is required e.g. to "neutralise" the red colour in the cyan positive, this could be done by printing the *magenta*† positive a little less heavily where it corresponds with the cyan image. A low-contrast positive transparency is therefore made from the cyan-printing negative and bound up in register with the magenta-printing negative for the printing operation, thus effecting the required diminution of magenta.

7. THE PROBLEM OF SOUND REPRODUCTION IN COLOUR CINEMATOGRAPHY

Sound reproduction presents a particular problem in the use of colour in professional cinematography. Normally, a tungsten-filament lamp is used, and the light it throws on a photocell is modified by variations in the amount of silver present in the sound track, the electrical impulses thus generated being amplified to operate the loud-speaking equipment. The photocells used have a maximum sensitivity in the near infrared, where the maximum emission of the lamp lies, and this is not transmitted by the silver grains. The dyes of a colour film, however, are transparent to these wavelengths, and the visible regions, which they absorb, do not affect the photocell to the same extent, so that a colour film, to give the same volume of sound, would require greater amplification, leading to increased noise background.

The solution to this problem is found either in modification of the film, e.g. selective removal of the silver from the picture space alone, and not from the sound track, by mechanical arrangements in the bleaching bath or by reconverting the sound-track dyes to a silver image; or it can be done by using a different type of photocell. A type of photocell which has maximum sensitivity in the visible region has been developed [59]. If the colour track is retained and the modified photocell employed, then it is best to print the sound record on a monopack film by blue light, which affects only the top layer and so minimises any risk of light scatter in the lower emulsions, which would deteriorate the sound quality.

III—The Chemistry of Colour Image Formation

In additive reproduction processes the images are formed in silver and so remain, the colours being determined by the choice of dyes for the filters, a choice based mainly on physical principles. In subtractive reproduction processes, on the other hand, the positive silver images must be transformed by chemical reactions into images in transparent colours. The colour images may be formed by a number of general methods, classified as follows—

(1) Transformation of the silver image into an inorganic coloured compound (*chemical toning*).

(2) Dyeing of the image after changing it into a form which may act as a mordant (*dye toning*).

(3) Localised destruction or formation of a dye by catalytic action of the silver image (*silver-dye-bleach* processes).

(4) Destruction of a dye by the action of light (*dye-bleach* process). This does not involve the preliminary formation of a silver image.

(5) Formation of dyes by reaction of oxidation products of a developer, with or without a second reagent present, at the points where image development takes place (*colour development*).

(6) Reproduction of silver images by duplicates in dye or pigment in layers of gelatin, later superposed by transfer on to a single support (*tanning* processes).

(7) Development to give images in coloured colloidal silver.

1. CHEMICAL TONING PROCESSES

This is the designation usually applied to methods by which the image is converted into a coloured inorganic or mixed inorganic–organic compound, other than by the use of dyes [51, 139]. The change may be effected directly without the intermediate use of silver compounds, as in the well known blueprint process, or, more often, by first producing a silver image, which is later converted into a form capable of reaction to produce coloured compounds. Many such reactions are based on the versatile behaviour of silver ferrocyanide, which can readily be formed by the action of potassium ferricyanide on the silver image—

$$4Ag + 4K_3Fe(CN)_6 = Ag_4Fe(CN)_6 + 3K_4Fe(CN)_6$$

Potassium ferrocyanide, being water-soluble, is removed by the processing solution. The silver salt is insoluble and remains in the emulsion, so that it can then be made to undergo a number of reactions to produce insoluble coloured products *in situ*.

(*a*) *Blue and Cyan Images*

By acting upon the silver ferrocyanide image with ferric salts Prussian blue is formed. Ammonium ferric citrate is usually employed, and

* The magenta and yellow images have common red transmission, so that the use of yellow here is equivalent to green. A suitable green compound would presumably be more difficult to synthesise.

† Since, of course, magenta and cyan have common blue transmission, variation in magenta is equivalent to variation of red only.

is dissolved in the initial potassium ferricyanide treating bath, so that toning is effected by a single solution. The use of the citrate is necessary because citrate radicals form largely unionised complexes with ferric ions, and thus the precipitation of ferric ferricyanide in the bath is prevented.

If a greener tone is desired, a treatment of the blue image with vanadium chloride solution may be given, to convert part of the silver ferrocyanide to a yellow vanadium ferrocyanide*.

(*b*) *Yellows*

Several methods have been suggested for the production of yellow images by chemical toning, of which the following are typical—

(i) Conversion of the silver image into silver ferrocyanide by means of potassium ferricyanide solution, followed by treatment with vanadium chloride to give yellow vanadium ferrocyanide.

(ii) Treatment of either a silver bromide or a lead ferrocyanide image with a solution of potassium iodide and mercuric chloride.

(iii) Use of uranium compounds to produce a variety of reddish and yellowish tones, according to the particular conditions and reagents used, the reaction again taking place with the intermediate formation of silver ferrocyanide. The image may e.g. be treated with a solution containing uranyl nitrate, ammonium oxalate, potassium ferricyanide, and hydrochloric acid.

(iv) Formation of cadmium sulphide—The silver image is bleached in a solution containing cadmium nitrate, potassium citrate, citric acid, formaldehyde, and potassium ferricyanide, silver ferrocyanide remaining in the bleached image. The print is then temporarily removed from the solution while sodium thiosulphate is dissolved in the latter, and then returned. The thiosulphate dissolves out the silver ferrocyanide, but just as this occurs (provided the conditions are correctly controlled) the ferrocyanide is reprecipitated as cadmium ferrocyanide. After washing, the cadmium ferrocyanide image is converted into sulphide by a solution of sodium sulphide and sodium bicarbonate.

(*c*) *Magenta*

(i) Copper ferrocyanide is red-brown, and a silver image may be converted thereto by treatment with a solution containing copper sulphate, potassium citrate, and potassium ferricyanide.

(ii) Mixed organic-inorganic compounds have also been used for magenta tones, e.g. the products of the reaction of silver salts and *p*-dimethylaminobenzylidenerhodanine, or nickel dimethylglyoxime. The latter can be formed by treating the silver image with a solution of nickel nitrate, potassium nitrate, potassium ferricyanide, and nitric acid, and then, after washing, with a solution of dimethylglyoxime and sodium hydroxide.

The chemical toning methods have now been largely outdated by the processes using dyes, because these have greater flexibility and enable a much wider range of tones to be obtained. One commercial process* has been made available, however, for producing subtractive paper prints by a transfer process, utilising separation images of Prussian blue, nickel dimethylglyoxime, and cadmium yellow.

* Presumably some silver ferrocyanide must remain after the formation of Prussian blue.

2. DYE TONING PROCESSES

The silver image may be converted into a coloured image by first transforming it into a compound with mordanting properties for dyes, usually of the basic class, by conversion into any one of a number of types of derivatives—e.g. (*a*) silver halides, especially silver iodide, either alone or as complexes with organic reagents, (*b*) various ferricyanides and ferrocyanides, (*c*) complex compounds containing silver and chromium, (*d*) thiocyanates, (*e*) silver sulphide†, (*f*) ferric hydroxide, and (*g*) titanium dioxide. It may also be made into (*h*) silver-diazo complexes, from which azo dyes can be formed.

(*a*) *Silver Halides*

Of these, silver iodide has been found most successful. It may be formed by treating the silver image with an oxidising bath containing iodide ions [146], but the silver iodide thus formed is opaque and, after the dye has been fixed with tannic acid, must be removed to improve the transparency. If the dyed image, however, is treated with a 20% solution of potassium iodide, the silver iodide is rendered transparent and need not be removed [133]. A further simplification of procedure is to oxidise with an aqueous solution of iodine and acetic acid containing a high concentration of potassium iodide. This forms a completely transparent complex potassium silver iodide image, which adsorbs considerably more dye than does the opaque mordant. The gelatin must first be hardened with formaldehyde to overcome the softening effect of the treating bath [15,16]. This treatment produces so effective a mordant that special developers must be employed to give only a weak, though correctly graded, silver image. The process has been examined for large-scale application in colour cinematographic printing. Suitable dyes [51] are Astraphloxine FF, Rhodamine B (*C.I.* 749), or Rhodamine 6G (*C.I.* 752) for magenta; Rhoduline Sky Blue, Setapolin (*C.I.* 663), Thionine Blue (*C.I.* 920), or Methylene Blue (*C.I.* 922) for cyan; and Auramine (*C.I.* 655) or Thioflavine T (*C.I.* 815) for yellow.

Thiourea [22] and other thioamino compounds [148] may be used to form transparent complexes with silver images, which have first been converted to halide. These are said to have good mordanting properties.

A brown image, possibly containing a silver perbromide, having mordant properties for basic dyes, can be formed by the action of an aqueous solution of benzoquinone and potassium bromide on a silver image [126].

(*b*) *Ferricyanides and Ferrocyanides*

The gelatin layer containing the silver image may be treated with a basic dye solution, whereby

* Chromatone (Defender Photo Supply Inc., U.S.A.).

† Reported to act as a mordant only if impure.

the dye is adsorbed; then by treatment with a solution of chromic acid and potassium ferricyanide, silver ferricyanide is formed and simultaneously fixes the dye [58].

In a related process stable leuco compounds of basic dyes are incorporated in the emulsion, and after development, fixation, and washing, a solution of chromic acid, copper salts, and ferricyanide is applied. This oxidises the leuco compounds, which are simultaneously fixed by becoming mordanted upon silver copper ferricyanide [3].

Silver ferrocyanide itself is not satisfactory, but it can be converted into more efficient mordants composed of ferrocyanides of other metals, e.g. tin (stannic), copper (cupric), or double ferrocyanides of silver and e.g. cobalt, copper, iron, lead, or uranium*, all of which act as mordants for basic dyes with different degrees of efficiency.

(*c*) *Complexes containing Silver and Chromium*

Mordant complexes of silver and chromium are formed by treating a silver image with a solution containing potassium ferricyanide, chromic acid, and acetic acid [79]. The reduction products of potassium dichromate itself, formed in gelatin on exposure to light, are unsatisfactory as a mordant.

(*d*) *Thiocyanates*

A colourless thiocyanate complex of copper and silver, with good mordanting properties for basic dyes (and acid dyes of phthalein type), is formed by treating a silver image with a solution containing copper sulphate, potassium thiocyanate, potassium citrate, and acetic acid [23]. This mordant can be made almost invisible in the gelatin before the dye is applied.

(*f*) *Iron and* (*g*) *Titanium*

Ferric hydroxide, suitable as a mordant, can be formed in place of the image by first transforming to Prussian blue and then treating with alkali.

By developing the exposed emulsion with titanous oxalate in place of the usual organic developers, an image containing both silver and titanium dioxide is said to be formed, which has mordanting properties for both acid and basic dyes [122].

(*h*) *Mordants for Azo Dyes*

Azo compounds may be formed on the image by a number of methods, e.g.—

(i) A diazonium solution is applied to a mordant image containing cuprous iodide and then coupled with a "naphthol" [110]. Suitable combinations are formed by using diazotised *p*-aminodiphenylamine, coupled (in alkali) with J acid for magenta, H acid for cyan, and *m*-cresol for yellow.

(ii) The silver image is converted to a silver *anti*-diazoate, using silver chloride as an intermediate. The *anti*-diazoates are used in preference to the *syn* forms to prevent coupling with the gelatin itself. Treatment with a coupling component at a controlled pH gives an azo dye corresponding with the original silver image [69]. Alternatively, an aromatic amine which forms an insoluble silver salt, e.g. 6-amino-2-mercaptobenzothiazole, may be applied to a silver bromide image, and later diazotised and coupled.

(iii) The silver image is transformed into complex nitrites, which react with amines to give diazonium compounds, from which azo dyes are formed. Thus the image may be treated first with thallic chloride to form thallous chloride and silver chloride, and then with sodium cobaltinitrite to form insoluble thallium cobaltinitrite complexes, which, on treatment with acid, release nitrous acid [96].

Clark [24] studied the mordanting properties for acid and basic dyes of suspensions of insoluble ferri- and ferro-cyanides and halides. Halides of silver, manganese, cobalt, nickel, and iron were found to have poor mordanting powers; those of bismuth, cadmium, cerium (-ic), copper (cuprous and cupric), tin (stannous and stannic), vanadium, and uranium had improved properties; zinc and the remaining heavy metals, e.g. mercury and thorium, gave good results. Ferricyanides were found to be better mordants than ferrocyanides. Both types of salt act as mordants for basic dyes better* than for acid dyes, when the anion is present in slight excess.

Seyewitz [126] has published a classified list of dyes showing their relative toning efficiencies.

Bullock [19], who discussed the theoretical aspects of dye toning, believed that the mordanting action is due to adsorption of the dye by oppositely charged ions adsorbed on colloidal particles in the emulsion. For example, copper ferrocyanide loses most of its mordanting power for basic dyes if adsorbed potassium ferrocyanide or ferricyanide is washed out, and regains it on retreatment with either. On the other hand, basic dyes have less, and acid dyes more, affinity for gelatin when it contains colloidal aluminium hydroxide (believed to be positively charged) than when it does not.

3. SILVER-DYE-BLEACH PROCESSES†

These processes [52] depend upon the ability of the silver image to catalyse the destruction or formation of a dye by oxidation or reduction. This catalytic activity has been known for some considerable time. Thus, a process for making colour prints by utilising the already known catalytic activity of silver on hydrogen peroxide was suggested in Schinzel's original integral tripack disclosure of 1905 [51]. Each layer in the pack was sensitised to one of the primaries, and incorporated an appropriate complementary-coloured dye, unaffected by the normal processing baths. After exposure and development, a treatment with hydrogen peroxide would destroy the dye by oxidation at the points where the silver images were present.

The corresponding catalysis of reduction processes was observed later [23]. Christensen in 1918 discovered that some dyes are reduced by dithionite

* Uranium mordants, because of their colour, are suitable only for orange-red images, such as are necessary in two-colour processes.

* Except lead and thorium ferrocyanides.

† This section is largely based on the admirable survey of these processes given by Friedman [52] (*op. cit.*, Chapter 24).

(hydrosulphite) or stannous chloride, at room temperature, only in presence of finely divided silver. A dye of this type could be incorporated in the emulsion in manufacture or applied after development, and so reduced. Several other workers have made contributions to the subject, but credit for its intensive development must go to Gáspár[52], in whose name stand a very large number of patents covering diverse elaborations of the basic principle*. The following paragraphs give a summary of some of the more important reactions covered by these inventions.

A dye which does not readily form a leuco compound is uniformly dispersed throughout an emulsion, and is subsequently destroyed at the points containing the silver image, by either oxidation or reduction[52 a–e, g]. Azo acid and direct cotton dyes may be used, and also a few from other classes, e.g. anthraquinone or basic compounds. Reagents may be used which attack the dye even in absence of silver, since the metal accelerates the reaction, or compounds may be employed which are inactive in absence of silver. These are believed to form silver salts which act upon the dye either directly or by the formation of intermediate products, the amount of dye destroyed thus being proportional to the amount of silver.

Examples of suitable reducing reagents are—sodium sulphide and metol; thiodiglycollic acid and sugar; semicarbazide, thiosemicarbazide, guanidine, or aminoguanidine (in dilute sulphuric acid); and thiourea and citric acid. Developer solutions may also be used which reduce the dye during the actual developing process, e.g. a solution containing amidol, sodium bisulphite, and sodium sulphite[52c].

Examples of suitable oxidising agents[52c,d] are—sodium chlorate and hydrochloric acid; or potassium bromate, potassium iodide, and sulphuric acid, with copper sulphate or vanadium chloride as catalyst. In this case, the dye is destroyed only where the *undeveloped* silver salts are present. When a solution of bleaching powder and acetic acid is used it is destroyed at the positions occupied by the silver image.

Stable derivatives of leuco vat dyes may be introduced into the emulsion in manufacture, and oxidised to the vat dyes later at the points occupied by the silver image, e.g. by a step-by-step procedure involving the successive formation of lead ferrocyanide, lead chromate, and chromic acid[52c] or by means of ammonium persulphate[66].

The reactions are accelerated by certain catalysts. Thus, quinol (hydroquinone) or metol in aqueous ethanol may be used with thiourea[52f], or compounds of general formula[52a]—

(X, Y = N, S, or O), e.g. Safranine (*C.I.* 841), Phenosafranine (*C.I.* 840), or Methylene Blue (*C.I.* 922). Such compounds bleach readily in light, especially in presence of thiourea or its derivatives, to form leuco compounds which have strong reducing properties, and thus they presumably act as hydrogen carriers. Gáspár believes the general reaction to be represented by—

$$\text{Ag} + \text{H}^+\text{X}^- \rightleftharpoons \text{Ag}^+\text{X}^- + \text{H}$$

Normally this proceeds in the forward direction, but reversal may occur in presence of a substance capable of removing silver ions from solution. The reaction, e.g. of thiourea on an azo dye in presence of acid and silver, may be formulated thus—

$$4\text{Ag} + \text{R}^1\text{·N:N·R}^2 + 4\text{HCl} + 8\text{CS(NH}_2)_2 \rightarrow \text{R}^1\text{·NH}_2 + \text{R}^2\text{·NH}_2 + 4\text{Ag[CS(NH}_2)_2]_2\text{Cl}$$

Friedman[51] suggests an alternative mechanism, based on the adsorption theory of development, to explain several variations of the general reaction, which are not so readily accounted for on Gáspár's hypothesis. He suggests that the metallic specks forming the latent image may adsorb ions of the developer, e.g. metol, and thus raise its concentration to a point where its reducing power becomes high enough to develop the image; at this stage, the dye may also be reduced.

Many methods are proposed for producing a uniform dispersion of non-diffusing dye in the gelatin layers. The following are examples—

(*a*) Incorporation of the water-soluble sodium salt of the hydrazo form of a Naphtol AS dye in the emulsion, followed by its oxidation by e.g. nitrous acid or ferric chloride to the insoluble dye.

(*b*) Use of water-soluble bisulphite compounds of insoluble azo dyes with subsequent regeneration of the original colouring matter.

(*c*) Coupling of an *o*-sulphonic acid derivative of an amine with a diazo compound to form a soluble dye, which, after dissolution in the gelatin, is precipitated as a fine dispersion by the addition of alkali.

(*d*) Formation of an amine from a nitro or nitroso compound in the emulsion layer[52g]. The reduction can be effected by hydrosulphite after the development, fixation, and washing are complete. Diazotisation and coupling, and then bleaching follow.

(*e*) Instead of a general reduction of the amine as in (*d*), a reduction only at the points occupied by silver can be effected by treatment with e.g. a solution of thiourea and hydroquinone in dilute sulphuric acid. Diazotisation and coupling then give a dye image corresponding to the silver one.

(*f*) Formation of inorganic pigments, e.g. by—

(i) Dissolving ferrous salts in the gelatin, and then treating with potassium ferricyanide.

(ii) Dissolving a soluble lead salt in the gelatin, and then treating with a soluble dichromate. The Prussian blue and lead chromate so formed can be reduced in presence of the silver image by one of the methods used for dyes.

(*g*) Incorporation of soluble dyes in the gelatin, followed by precipitation of their insoluble salts with bases, e.g. guanidine or quinoline derivatives[52h,i,j].

* Prior to the introduction of the present systems of colour development, the I.G. appear also to have investigated these processes.

(*b*) Incorporation of dyes of the Neolan (Ciba) type without further treatment, e.g. Neolan Yellow R, Neolan Green BR, and Neolan Pink G for the three minus primaries [52b].

Alternatively the precipitants may be incorporated in the gelatin, and subsequently treated with the dye [52*l,m,n*]. A complex formed with one dye may not necessarily interfere with the diffusion of a second dye, which can then penetrate below the first complex and so give a "layered" effect in the emulsion.

The above are typical of the general principles of coloured image formation in the silver-dye-bleach process. Problems arise, of course, in their actual application to colour photography. The principal value of these reactions lies in the possibility of their use for the formation of three-layer integral monopacks. For negative monopacks it is not satisfactory to incorporate the actual dyes in the layers, because of their light-filtering action. The top layer, blue-sensitive and therefore necessarily coloured yellow, would be insufficiently exposed because of the absorption by the dye of blue light in its passage through the emulsion*. It is therefore preferred to use colourless components which are formed into dyes after development of the image. If one layer is coated on the opposite side of the base to the other two, the difficulty may be overcome, though with loss of definition. An arrangement suitable for printing from opposite sides with red and blue lights consists of this sequence—

Magenta-dyed, blue-sensitive layer
Yellow-dyed, red-sensitive layer
Film base
Cyan-dyed, blue-sensitive layer

For positive film, the same restriction does not apply, because the original master negative may be coloured in shades not necessarily bearing any relation to those of the original subject. This enables a choice of printing lights to be made for the positive emulsion which will not be interfered with by the dyes present [52*a–e*]. For example, the layers may be arranged in a positive monopack thus—

	Sensitivity Region	Dye	Print from Separation Record
	Green	Yellow	Blue
Direction	Red	Magenta	Green
of light	Infrared	Cyan	Red
↓		Base	

A little consideration will show that each layer is reached only by the particular coloured light to which it is sensitised, because, while all the dyes freely transmit infrared radiation, the cyan absorbs red, the magenta green, and the yellow blue light. Such a monopack is suitable for printing from individual separation negatives or from a coloured original.

Though the silver–dye-bleach process has been so intensively studied, it has not so far been widely employed commercially, one reason being that the film speed is low. Some cinematograph colour cartoon films have, however, been produced by its aid in Germany and the U.S.A.

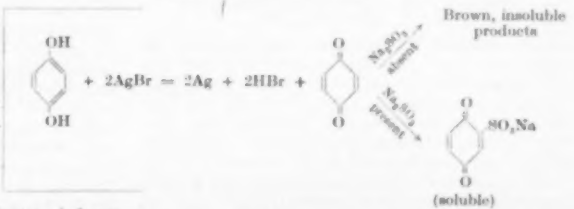
4. DYE-BLEACH PROCESS

A remarkable direct colour-printing paper, Utocolor, was introduced about forty years ago [95]. In principle, it depended on the direct bleaching of dyes by light. A single emulsion coloured with a mixture of dyes of the three primary colours was exposed under a colour transparency. Each dye is bleached only by the light it absorbs, in accordance with the well known Grotthus–Draper law, so that all the colours of the original are reproduced in the print after exposure, and the only further treatment needed is rinsing in water. The method has the merit of extreme simplicity and is theoretically most attractive. It is believed to have failed to gain support owing to the difficulty of ensuring regulated bleaching of all three dyes at once and possibly because of the extremely long exposures (1–2 hr.) required*. Erythrosine (*C.I.* 772, 773), Auramine (*C.I.* 655), and Methylene Blue (*C.I.* 922) were said to be suitable dyes, and were dispersed in a layer of gelatin.

5. COLOUR DEVELOPMENT PROCESSES

(*a*) *General Principles*

Development of an exposed silver image in normal black-and-white photography is usually carried out with a solution of an organic reducing agent, e.g. quinol (hydroquinone), *N*-methyl-*p*-aminophenol sulphate (metol), or 2:4-diaminophenol (amidol), in presence of sodium sulphite. If sulphite is omitted from the bath, the silver image is of a brownish colour instead of the usual black. This is due to the presence of insoluble, coloured oxidation products of the developer, formed in exact correspondence with the silver image, which are removed by the developing solution when sulphite is present. The reactions may be represented thus—



After development, the silver image may be dissolved out of the emulsion†, and thus, if the

* Gáspár has proposed [52*o*], however, to incorporate only small quantities of dye and to use a set of three emulsion layers on each side of the film base, each set being printed separately. The two sets of images combined give a satisfactory colour rendering.

* An ethereal solution of hydrogen peroxide was said to reduce this time to 10 min.

† Usually by a solution of sodium thiosulphate and potassium ferricyanide (Farmer's reducer).

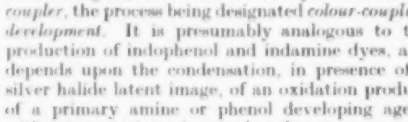
development has been carried out in absence of sulphite, an exactly corresponding image will remain formed of the organic oxidation product.

This procedure can be made the basis of methods of producing photographic images in the form of brightly coloured dyes, and one of these methods has become of great importance in recent years.

Two variations of the basic reaction have been studied. In one, known as *primary colour development*, a leuco derivative of an insoluble dye is used as developing agent, and after removal of silver a coloured image of the dye itself remains. Thus, indoxyl and thioindoxyl leave images of indigo and thioindigo respectively (Homolka 1907). The possibilities of this process are rather limited, and it is not used in practice.

(*b*) *Colour Coupling*

In the second, and currently used process, the oxidation product of the developer reacts with a second reagent, known as the *colour former* or *colour coupler*, the process being designated *colour-coupling development*. It is presumably analogous to the production of indophenol and indamine dyes, and depends upon the condensation, in presence of a silver halide latent image, of an oxidation product of a primary amine or phenol developing agent with an aromatic amine, a phenol, or a compound containing a reactive methylene group. The oxidation of *p*-phenylenediamine by silver halide in presence of phenol may be taken as an example—



Little investigation into the mechanism of colour development seems to have been carried out, and the reaction sequence may well be more complex than the above equation would indicate. Arbuzov [8] has demonstrated the formation of an azomethin dye when a pyrazolone coupling component is used.

Fischer and Siegrist [49] described the use of *p*-diethylaminoaniline (*NN*-diethyl-*p*-phenylenediamine) as the developer, and for the formation of the three minus colours they employed colour couplers of the following types—

Cyan	...	2:4-Dichloro-1-naphthol
Magenta	...	*p*-Nitrobenzyl cyanide
Yellow	...	Ethyl acetoacetate

These workers described a large number of developers and couplers (Table III), and so thoroughly did they cover the ground that, in spite of the very great research activity in this field over the past fifteen years, few fundamentally new types of compound have been described*, and many in use to-day are based on their disclosures, except that, as substances with reactive methylene groups, pyrazolone derivatives have been used in place of benzyl compounds for the magenta colour. Indeed, the developing agent originally proposed by these workers (*p*-diethylaminoaniline)* is still widely used to-day.

TABLE III

Developers and Colour Couplers [49]

Developers	Couplers
Hydroquinone	α-Naphthol
p-Aminophenol	Xylenol
p-Aminophenol	α-Naphthol
Dimethyl-*p*-phenylenediamine	α-Naphthol
Dimethyl-*p*-phenylenediamine	Phenol
p-Phenylenediamine, *p*-toluylenediamine, amino-*p*-phenylenediamine, chloro-*p*-phenylenediamine, ethyl-*p*-phenylenediamine, *p*-aminophenylenepiperidine	Phenols, cresols, α-naphthol, *o*-aminophenol, trichloro-α-naphthol, resorcinol methyl ether, α-naphtholsulphonic acids, ethyl acetoacetate, malononitrile, chloroacetophenone, diketoindane, *p*-nitrobenzyl cyanide, thioindoxyl, thioindoxylcarboxylic acid, and chloroethyl acetoacetate
Diethyl-*p*-phenylenediamine, etc.	Thymol, 2:4-dichloro-1-naphthol, pentabromonaphthol, ethyl benzoylacetoacetate, dibenzoylmethane, ethyl cyanoacetoacetate, cyanoacetophenone, coumaranone

(*c*) *Colour Couplers for Incorporation in the Emulsion*

The incorporation of colour couplers in the emulsions very much simplifies the development process. Much effort has therefore been expended in devising means of preventing diffusion of couplers from one layer to another during development, so that they can be safely incorporated in the film during manufacture. Agfa has been active in pursuing this aim, and the Agfacolor films incorporate couplers whose molecules contain a bulky group which, being non-conjugated with the resonating system of the coupler molecule, does not affect the colour, but so decreases its solubility that the speed of diffusion becomes negligible. Examples of such groups are—alkyl chains containing more than five carbon atoms, hydroaromatic or hydrogenated heterocyclic residues, sterol residues, and carbohydrate residues [e.g. 71]. It has also been proposed to repeat the active part of the molecule many times, through non-conjugated links, by formation of a polymer with ethylene oxide [72]; or to replace the gelatin by film-forming polymers which themselves have colour-forming properties, e.g. polyamides or acetals of polyvinyl alcohol having colour-forming groups attached at points in the chain. This last process has, indeed, been commercialised [7, 72] in the du Pont Type 275 Color-release positive cinematograph film [116, 116, 117].

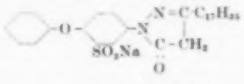
In actual fact, all the couplers used in Agfacolor films and paper contain a C_{17} or C_{18} group and a sulphonic or carboxylic acid group, which serves the double purpose of giving the molecule alkali solubility and anchoring it to the gelatin by salt-formation. They are finely dispersed by means of

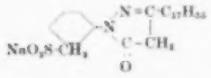
* A very large number of compounds for use in colour development have been patented in the past fifteen years or so, and the number continues to grow steadily. For full details reference should be made to patents granted principally to the following firms and their associates—Agfa Ansco, Dufay-Chromex, du Pont, Eastman Kodak, General Aniline, Gevaert, Ilford, ICI, I.G., and Kodak; and also to Dr. B. Gaspar.

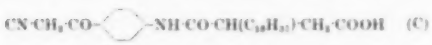
* *p*-Phenylenediamine compounds are notoriously liable to produce dermatitis. Schneider [123] found that the introduction of an *o*-hydroxy group, e.g. as in *p*-*N*-ethyl-*N*-hydroxyethylaminoaniline, considerably reduces this hazard without detriment to the developing properties.

wetting agents before mixing in the emulsion. The constitutions [1, 21, 120] are given below, and their use in Table IV.

MAGENTA COUPLERS

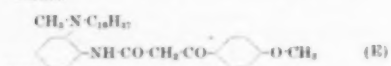

(A)


(B)

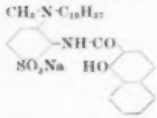

(C)

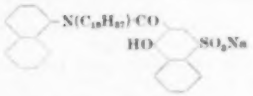
YELLOW COUPLERS

(D)


(E)

CYAN COUPLERS


(F)


(G)

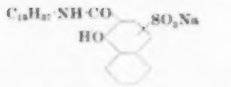

(H)

TABLE IV

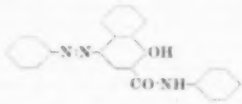
Arrangement of Colour Couplers in Agfacolor Films and Paper [21]

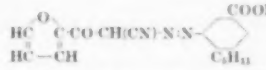
Film or Paper	Couplers: Magenta	Yellow	Cyan
REVERSAL			
Daylight	A	D	F + G
Half-watt light / Copy film ...	B	D	F + H
NON-REVERSAL			
Negative types for daylight, arclight, half-watt light ...	A	D	F
Positive	A	E	H
PAPER	C	D	H

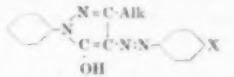
In the Kodacolor (negative) film and paper the couplers are rendered non-diffusing by a different principle. Each is dissolved in a resin which is dispersed as globules in the emulsion, so that silver halide particles and resin globules lie intermingled. Suitable natural or synthetic resins are e.g. gum mastic, gum dammar, polyvinyl acetate, and cellulose esters or ethers [93]. These are permeable to, but insoluble in, water.

(d) Coloured Colour Couplers for Automatic Masking

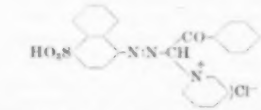
The purpose of these compounds has already been explained (§ II F 6(*b*)). Typical examples [44, 45, 54, 89, 90] are—


(I) [44]

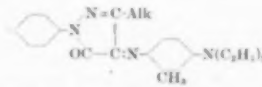

(II) [45]


(III) [89]

(X = H, O·CH$_3$, etc.)


(IV) [54]

Compounds of type I are yellow to orange-red for development to cyan; types II, III, and IV are yellow, for development to magenta. Compound III, e.g. on colour development with 2-amino-5-diethylaminotoluene hydrochloride, loses the azo group and is transformed to a compound similar to V—


(V)

The reaction mechanism has not yet been extensively studied. Vittum and coworkers [151] have put forward a mechanism based on the fission of the azo group in the coupler by reaction with a free radical formed from the developing agent.

(e) Processing Monopack Films

No particular difficulty arises in developing, with a colour-forming developing agent, negative–positive monopacks in which the colour couplers are in the emulsion, the normal development and fixation processes being employed (Fig. 4). On the other hand, the variety of reversal processes in which the colour couplers are applied along with the developer itself presents very great difficulties, and the manner in which they have been overcome represents an outstanding achievement.

The original Kodachrome developing process [43, 43, 91] used eight successive processing baths, and there were three interruptions for drying [104]. Three separate developing solutions were used, one for each colour layer, containing the appropriate colour couplers and reagents added to control the rate of penetration into the emulsions. The

exposed film was developed in a normal developer to give a negative in all three layers, bleached with permanganate to remove the negative silver images, exposed to light to enable the remaining silver bromide to be developed, and then colour-developed to produce a cyan in all three layers. It was then dried, and treated with a bleach capable of penetrating only the two top layers to remove the dye, the silver bromide then being regenerated and colour-developed to magenta; bleaching of only the top layer and a yellow colour development followed. A number of reagents have been suggested [83, 85a, 91] to aid control of penetration, either by retarding it by increasing the viscosity, e.g. sugars, cellulose derivatives, gelatin, gums, etc., or by accelerating it, e.g. ethylenediamine.

Modern processing methods avoid the cumbersome controlled-penetration procedure. The top and bottom emulsions are separately re-exposed to light prior to colour development, each from its own side, without affecting the middle layer; this is later separately re-exposed and colour-developed.

About 1940, Kodak began to use a process of this type for Kodachrome film [63, 66b, 99]. It involves only five baths and no intermediate drying. The sequence of operations is as follows [101]—

(i) Develop in an ordinary developer

(ii) Expose through the base to red light, which affects only the bottom layer, and renders developable the unexposed silver bromide therein

(iii) Colour-develop this layer to cyan

(iv) Expose the top of the film to blue light, which does not penetrate the yellow filter below the top layer

(v) Colour-develop to yellow

(vi) Expose the middle layer to green light

(vii) Colour-develop to magenta

(viii) Remove all the silver images and the yellow colour of the filter layer by means of potassium bromide–potassium ferricyanide bleach liquor.

Ilford [67, 67a, 67b] disclose related methods. Two barrier layers, one on either side of the emulsion layer, may contain a colourless surface-active agent to confer substantivity for a dye applied after the first development. Before re-exposure of the middle layer, this dye is washed out, preferably with the assistance of an oppositively charged surface-active agent. Alternatively, the barrier layers may contain (1) a small quantity of silver sulphide insufficient to colour the layer and (2) a very insensitive silver iodide–bromide emulsion. The upper of these filter layers also contains a yellow filter dye, which is bleached out in development. The initial development is made with a developer which develops the negative images in all three emulsions and also produces a yellow-brown deposit of colloidal silver in the barrier layers. This is opaque to blue light, but transparent to ultraviolet radiation. Thus the two outer emulsions can be re-exposed for colour development, from their own sides, in blue light, and then the middle emulsion can be exposed to ultraviolet radiation for separate colour development. The final bleach removes all silver, including that in the barriers.

(f) *Mechanism of Colour Development*

Doubts have several times been expressed regarding the correctness of Fischer and Siegrist's formulation [49] of the reaction sequence in colour development. Thus Tull [149] has shown that the supposed intermediate compounds, the oxidation products of the developer, do not themselves couple with the colour couplers, and he suggests that the latent image itself must be involved in the reaction. Flannery and Collins [50] have also shown that the dye molecules : silver atoms ratio in colour-developed emulsions does not agree with that required by the accepted reaction mechanism. Vittum and Brown [150] have proposed a mechanism involving reactions between ions of coupler, silver, and semiquinones formed from the developer. It would appear that there is much scope for further research into this problem.

(g) *Filter and Anti-halation Layers*

All monopacks must have a yellow filter layer below the top emulsion. This is coloured with colloidal silver*, which is removed along with the undeveloped silver during processing.

Behind the base there must be a coloured anti-halation layer to prevent reflection of light. A gelatin layer is used for flat films, and one of an alkali-soluble resin for 35-mm. films. In the Agfacolor films the following colouring matters were used for colouring this layer [21]—

Reversal films ...	Black colloidal silver
"Planfilm" ...	Acid Green and Acid Fuchsine
All other Agfacolor films	Green dye prepared by reaction of 3-amino-6-chlorobenzoic acid with dimethoxytriphenylcarbinol

There is also usually a plain gelatin separating layer between the middle and lower emulsions, and a top, outer protective ("anti-scratch") layer.

6. TANNING PROCESSES

(a) *General Principles*

The tanning (i.e. insolubilisation) of the gelatin image takes place by the action of either chromic salts or organic quinonoid compounds. The gelatin may be previously coloured by pigment or dye, and the unhardened portions washed away; or the hardened image or its unhardened counterpart may be used as a means of transferring dye to another support.

Three general methods of tanning may be defined—

Reduction of a soluble dichromate in a gelatin layer by (i) exposure to light, or (ii) the action of a silver image. In both cases simultaneous tanning action occurs.

(iii) Development of an exposed silver image in a sulphite-free phenolic developer, whereby the quinonoid oxidation products of the developer tan the gelatin.

Each of the above tanning methods may be used with one of three general methods of producing the final coloured positive—

(1) An insoluble or a soluble dye is incorporated in the gelatin before use, and after formation of the

* Dyes have been proposed for this purpose, but none so far discovered is satisfactory.

image in tanned gelatin, the untanned portions are washed away with hot water.

(2) The tanned gelatin image, after the untanned portions have been removed, is used as a matrix to absorb dye from a solution and transfer it to a fresh layer of gelatin.

(3) The whole gelatin layer, after tanning, but without removal of the untanned parts, is treated with an acid dye solution. The untanned parts are preferentially stained, and a transfer image may be made from them on fresh gelatin.

Methods (1) and (2) produce gelatin images in *relief*, the thickness varying with the extent of light action. In method (3) the surface of the gelatin is unchanged; hence this is said to form a *planographic* matrix. (2) and (3) are known as *dye transfer* or *imbibition* methods.

In all these print processes, very careful control of the relative contrast of the three images is essential to ensure good colour rendering, especially of white and neutral greys.

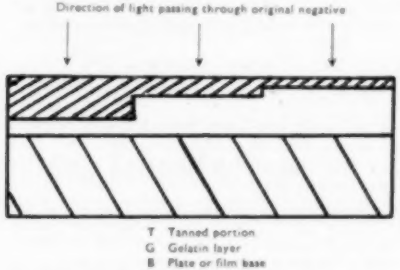


FIG. 25—Tanning of Gelatin on Exposure to Light

In the processes for producing relief images, it is essential for the gelatin of the image to vary in thickness in proportion to the light exposure. However, the light action starts at the outside and travels inwards (Fig. 25), and it is therefore obvious that, to obtain a true relief image, it is necessary either to transfer the gelatin layer to a fresh support, so that the hot water can attack the unaffected parts first, or to use a transparent base material and expose through the back. The addition to the gelatin of a soluble yellow dye (which washes out subsequently) helps to form a good relief image by retarding the penetration of the actinic (blue) light. (The emulsions are not colour-sensitised.)

Brief details of practical processes are given below, but for full information one of the text books on the subject e.g. [128] or the manufacturers' literature should be consulted.

(*b*) *Carbon and Carbro Processes*

(i) CARBON PROCESS—Images formed by exposing dichromated gelatin under a negative, and then washing in hot water, can be rendered visible by first incorporating carbon black or other coloured pigment in the gelatin (§ I 14). The gelatin layer may be transferred to a polished surface by squeegeeing when wet.

(ii) CARBRO PROCESS—By pressing a silver image against a pigmented gelatin layer previously sensitised in an acidified solution of potassium dichromate, potassium ferricyanide, and potassium bromide, tanning of the gelatin takes place at the portions in contact with silver. The sequence of reactions is believed to be somewhat as follows—ferricyanide–bromide solution diffuses into the silver emulsion, and there oxidises the silver to bromide; ferrocyanide thus formed diffuses back to the pigmented layer, where it reduces the dichromate, thus tanning the gelatin.

The Carbro process is an improvement on the carbon process, because the latter requires long exposures and can be used only for contact printing. With Carbro any previously prepared silver positive print, including enlargements, can be used.

The pigmented gelatin is supplied on thin paper, which, after the tanning operation, is squeegeed under water against a thin sheet of wax-polished celluloid or Perspex and allowed to dry incompletely. The paper is then removed, and the image treated with hot water (50°C.). All three separation positives are similarly treated, and are finally again transferred, but this time in superposition upon a permanent support of gelatin-coated paper.

(*c*) *Imbibition Processes*

(i) PRACTICAL DETAILS—In these processes, dye absorption and transfer can be repeated many times and prints can be obtained from the same original without the expense of using silver emulsions. This economy is a great advantage where the quantities involved are very large, as in the Technicolor cinematograph process. A brief summary of the salient features of these and related processes is given in Table V.

(ii) DISCUSSION—The imbibition process involves diffusion of dye in water from a *matrix* (master copy) to a gelatin surface. The process will steadily approach an equilibrium, which,

TABLE V

Transfer Processes for Prints

Process	Emulsion	Development†	Coloration
Carbro	Silver halide	Hot water	Dyed gelatin
Duxe-chrome [113]	Silver halide containing chrome alum*. Expose to artificial light.	Tanning developer (catechol) followed by hot-water etching.	Soluble dyes incorporated in emulsion during manufacture.
Wash-off Relief	Silver halide*. Expose to artificial light.	Metol–quinol developer, then wash, and tan in dichromate–sodium chloride–sulphuric acid; etch; set in cold alum solution.	Staining of gelatin relief matrix with acid dyes.
Pinatype	Dichromate–gelatin†. Expose to sun or arclamp.	Cold-water wash to remove unchanged dichromate.	Preferential staining of untanned gelatin (planographic).
Dye Transfer	Silver halide*. Expose to artificial light.	Tanning developer followed by hot-water etching.	Staining of gelatin relief matrix with acid dyes.

* A soluble yellow filter dye is also incorporated.
† Coated on glass plates.

however, is unlikely to be reached in the few minutes allotted in practice to the transfer stage. Immediately after transfer starts, dye will be absorbed by the copy equally from all parts of the matrix, so that the image will show no contrast

range at first. If the concentrations of dye in matrix and receiving layer at equilibrium are equal, then it is a simple matter to show that the different thicknesses of the matrix will be reproduced by differences in concentration of dye in the copy, but with a reduced degree of contrast. For example, if the tanned portions shown in Fig. 25 form the matrix image, of thickness 1·0, 0·5, 0·25 unit respectively, containing dye at unit concentration, then, when this is placed against a receiving surface layer (of uniform thickness, say 1·0 unit), transference takes place. At equilibrium the relative concentrations of dye in the receiving layers are—

$$\frac{1}{1+1}=\frac{1}{2} \qquad \frac{0{\cdot}5}{0{\cdot}5+1{\cdot}0}=\frac{1}{3} \qquad \frac{0{\cdot}25}{0{\cdot}25+1{\cdot}0}=\frac{1}{5}$$

The optical densities will be in the same proportion; in the original relief they were, however, in the ratio—

$$1:\tfrac{1}{2}:\tfrac{1}{4}=\tfrac{1}{2}:\tfrac{1}{4}:\tfrac{1}{8}$$

representing a wider range of contrast. Thus, starting from zero, the image contrast will build up steadily to a maximum at equilibrium. The above situation will, however, be complicated to an unpredictable extent by differences in diffusibility of the dyes used in the two forms of gelatin gel, tanned and untanned, if printing is from a tanned relief. In the actual working of the process, contrast can be increased by rinsing the relief, after dyeing, in dilute acetic acid, or decreased by a treatment in sodium acetate buffer solution. The acid may act by increasing the affinity of untanned gelatin for dye relative to the tanned portion, or by accelerating diffusion and so bringing the system nearer to equilibrium in the normal working period.

Acid dyes differ considerably in their relative absorbability by tanned or untanned gelatin from cold aqueous solutions under the conditions of the normal transfer process. They may be preferentially absorbed by one or the other, or absorbed by neither [109], for the following reasons—

The final result must depend upon both the ease of absorption of dye from the staining bath and the ease of washing out. Tanning will reduce swelling and ease of absorption, but it may also reduce the rate of loss of dye in the subsequent washing process. It is unlikely that either staining or washing out is carried as far as equilibrium under normal conditions, so that the final relative depths of shade on tanned and untanned portions will depend on the relative velocities of transfer of dye into and out of the emulsion, and these cannot readily be predicted.

Little appears to have been published upon the theoretical aspects of the action, or on the relation between dye constitution and suitability for transfer. Friedman [51] gives the following as suitable dyes—

MAGENTA	Alizarine Rubinol R (*C.I.* 1091) Acid Magenta 2B
YELLOW	Tartrazine O (*C.I.* 640) Fast Wool Yellow 3GL (*C.I.* 636)
CYAN	Wool Green B (*C.I.* 737) Patent Blue conc. (*C.I.* 712)

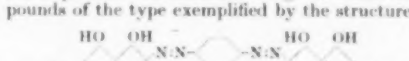
All of these are acid dyes.

In the Duxochrome process *non*-diffusibility is required, and so the chosen dyes are all of the direct cotton class, except the filter dyes, which must wash out. (Direct cotton dyes are in general less diffusible than acid wool dyes.) The dyes used are given [119] as—

MAGENTA	Brilliant Violet 5R
YELLOW	Chlorantine Fast Yellow Diphenyl Yellow Pure (+ a little Chlorantine Fast Green)
CYAN	Light Blue (*C.I.* 520) Diphenyl Blue Green Pure

The "wash-out" (filter) dyes are Neococcin for the yellow emulsion and Pure Yellow F4G for the other two.

The spectral requirements for dyes in subtractive print processes are the same, of course, as those for colour-development dyes, and again the difficulty is met of finding a cyan dye which does not transmit red. Efforts are being made to synthesise dyes of improved red absorption, and a recent patent [135] claims that blue–green disazo compounds of the type exemplified by the structure—



have the required broad and strong absorption band at the red end of the spectrum.

(iii) THE TECHNICOLOR PROCESS—In the Technicolor process the separation negatives in 35-mm. cinematograph film are processed to give a gelatin relief image*, and imbibition prints on clear gelatin stock are prepared by a process resembling the dye transfer method, except that to increase the number of copies without damage to the original, intermediate positives and negatives are produced. Exact working details of the technique used are not published, but the processes are covered by many patents *e.g.* [78, 134–140]. One disclosed developing solution contains pyrogallol and sodium hydroxide, without sulphite, and a described flow-sheet of the process runs as follows—

Original negative relief matrices from colour-separation negatives
↓
Auxiliary positive "key" records
↓
Auxiliary "key" negatives
↓
Separation (colour) positives
↓
Final three-colour positive

The solution of the problem of accurately registering the small images and the successful operation of such a complex process on the required mass-production scale are a considerable achievement.

(iv) PANCHROMATIC RELIEF FILM—This is a recently introduced material which enables three colour-separation positives to be made direct from a colour transparency (negative or positive). Red, green, and blue filters are used in exposing the

* Both tanning development and processes based on hardening with chromium compounds are included in the patents.

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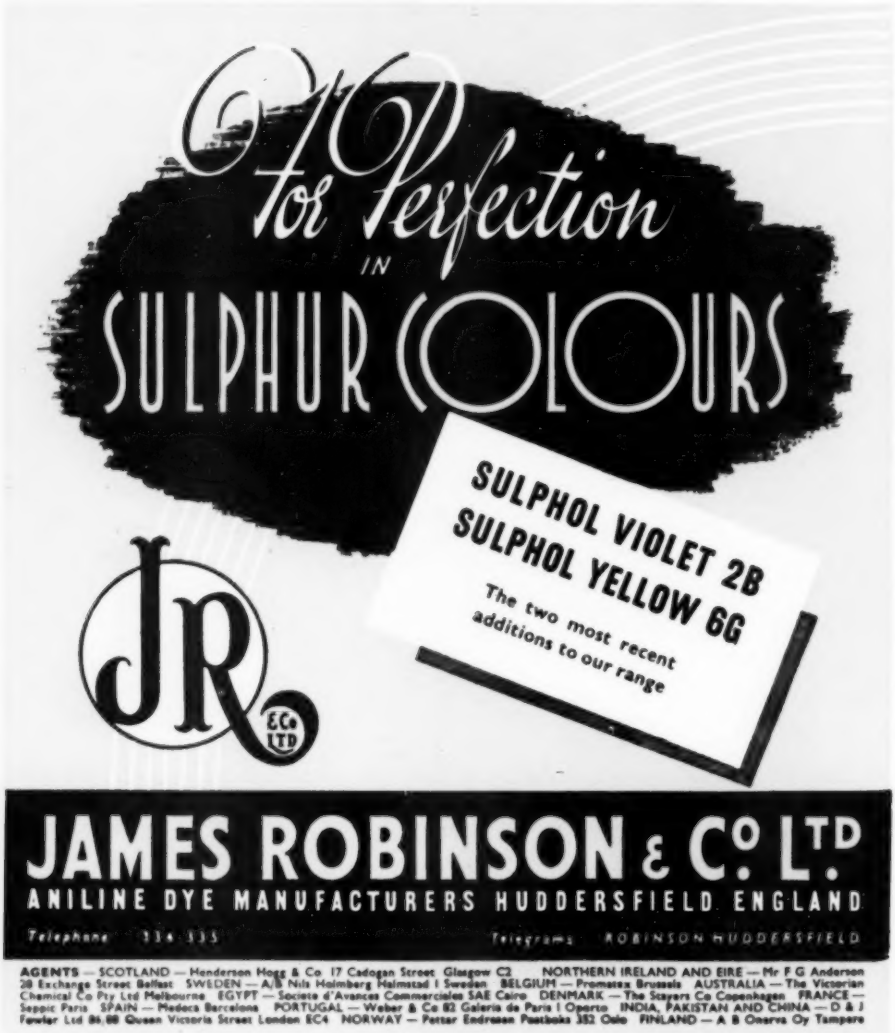

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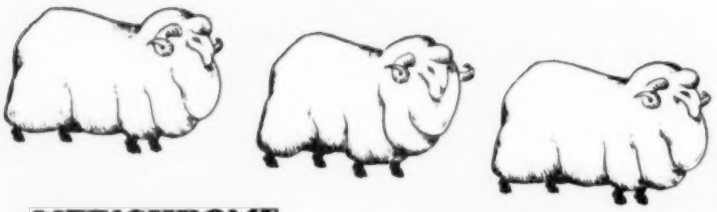

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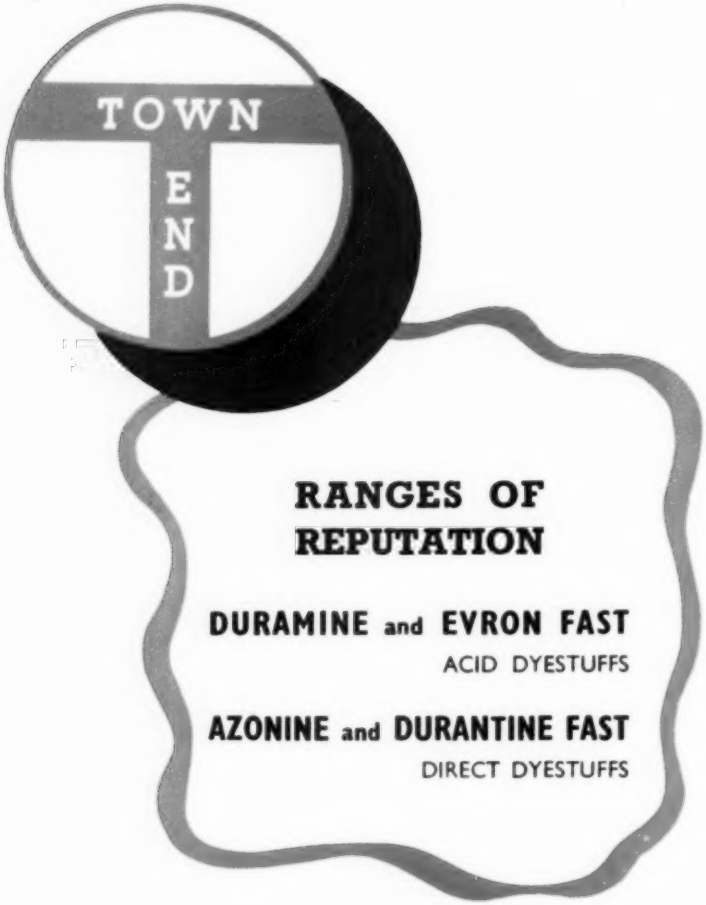

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three records, which are processed by tanning development and imbibition printing. If the original transparency is a negative, the procedure is exactly as already described; if it is a positive, the separation images produced are treated with hydrogen peroxide instead of hot water. This removes the *tanned* gelatin and leaves the untanned. Thus the imbibition prints will still be positives.

7. COLLOIDAL SILVER PROCESS

Occasional attempts have been made to obtain direct colour rendering in ordinary black-and-white silver emulsions, the range of colours being produced by variations in the colloidal state of the developed silver alone. Bensley [115] has recently made two-colour (subtractive) positives by such a method, using physical development.

IV—Conclusions and Appendixes

Until the introduction of the modern popular processes some fifteen years ago and the wide use of colour in the cinema, colour photography was almost unknown to the general public, but its popularity is now steadily increasing. With its present commercial importance and the work of the large industrial research organisations engaged in its development, we may expect an increasing pace of progress in the future*. It would be unwise to predict the direction of this progress very far ahead, for an unforeseen discovery may at any time reorient the lines of current research.

Nevertheless, present indications do suggest that, with improvement in colour rendering, negative–positive monopack processes will eventually replace both the one-shot camera and three-colour separation printing. Mosaic-screen material may continue to have considerable use because of its relatively low cost, the simplicity of the processing methods, and the attractive results it can produce.

For standard-size cinematography three-colour imbibition printing is well established because of its advantages of economy, but the ideal negative material is undoubtedly a monopack, and we may expect the replacement of the present cumbersome three-colour separation cameras by normal equipment carrying monopack film.

Finally, it may be well to point out that most of the fundamental inventions in colour photography originated in the creative imagination of isolated inventors, but that their successful practical application has been achieved only by the work of large industrial research organisations. This particular applied science therefore points a moral to our modern civilisation—creative technology needs both the unconfined imagination of the individual inventor or discoverer and the organised investigation of a team of specialists.

Owing to the impracticability of fully describing in this review all the processes of colour photography which have reached commercial development, and for convenience in reference, an alphabetical list of many of the better known commercial processes, both historic and modern, with brief notes, is given (Appendix A). This is based on similar ones given by Klein [81] and Spencer [128], with additions.

* * *

We wish to thank the many individuals and organisations who have helped us to compile this review. These include—the Comptroller of H.M. Stationery Office, Dr. D. A. Spencer, and Dr. W. D. Wright for permission to reproduce Fig. 8, 15, 19, 23; 4, 10, 13; and 17, 21 (in part) respectively; Dufay-Chromex Ltd., Ilford Ltd., and Sir Isaac Pitman & Sons Ltd. for the loan of blocks for Fig. 9, 11; 2, 12; and 4, 13 respectively; Mr. F. A. Kelly and Miss B. M. Light for typing the manuscript in its several stages of completion; Mr. I. S. Shaw for drawing the line diagrams; Mr. E. R. M. Milne and Dr. F. J. Symon for helpful discussion and criticism of the manuscript; Dr. G. B. Harrison and Prof. P. D. Ritchie for their interest and encouragement; and finally, the several authors, mentioned particularly in the Bibliography, from whose works we have freely drawn information.

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APPENDIXES

A—Commercial Name Index

Agfacolor—1. Mosaic-screen plate (1912)
2. Lenticular film for cinematograph and miniature still photography (1932)
3. Monopack reversal film (1936)
4. Negative–positive film and positive paper monopack (1943)

Anscocolor—Similar to Agfacolor (1943)

Aurora plate—Mosaic irregular-screen plate (ca. 1909)

Autochrome—Mosaic irregular-screen plate (1907–1933); film (1935)

Bassani—Additive cinematograph process using beam-splitting device

Belcolor—Subtractive printing process using superposed emulsions on transparent base

Brewster—Two-colour subtractive bipack cinematograph process (1914)

Busch—Two-colour additive cinematograph process with two-lens system (ca. 1928)

Carbro—Transfer print process with pigmented gelatin

Caron—American process resembling Carbro

Chemicolor—Two-colour subtractive cinematograph process

Chromatone—Transfer printing process using inorganic toning

Cinecolor—1. Two-colour subtractive cinematograph process, with beam-splitting camera
2. Two-colour additive cinematograph process
3. Three-colour subtractive cinematograph process

*The rate of growth of interest in the subject is indicated very roughly by the relevant patents listed in *Chemical Abstracts* during the periods 1917–1926 (108), 1927–1936 (693), 1937–1946 (1226), and 1947–1951 (244).

Colloprint—Transfer printing process
Colorcraft—Two-colour subtractive cinematograph process (prints dye-toned on iodide mordant) (ca. 1929)
Colorsnaps—Roll-film separable tripack for amateur use (ca. 1929)
Colorstil—American name for Duxochrome

Diachrome—Silver iodide–basic dye mordant transfer printing process (1905)
Dioptichrome—Regular mosaic processes, with screen (*a*) separable or (*b*) inseparable. Four colour elements (ca. 1916–1917)
Diopticolor—Early name for Dioptichrome plate
Dufay—Four-colour regular mosaic-screen plate (1908)
Dufaycolor—1. Regular mosaic-screen film (1934)
2. Negative-positive cinematograph process (1936)
Dufaytissue—Printing process resembling three-colour carbon (1941)
Dugromacolor—Three-colour additive cinematographic process using beam-splitting device (1948)
Dunning—Two-colour subtractive cinematograph process
Dupack—Separable bipack two-colour cinematograph process (du Pont, ca. 1931)
Duplex—Separable mosaic-screen plate (ca. 1927)
Duxochrome—Three-colour transfer printing process, using dyed gelatin emulsions (J. Herzog & Co., Bremen, ca. 1941)
Dye Transfer—Three-colour imbibition print process (Kodak 1946)
Dyebro—Similar to Carbro, gelatin reliefs dye-stained and not pigmented

Ektachrome*—Subtractive monopack reversal film (Kodak 1946)
Ektacolor*—Subtractive monopack negative film with automatic masking couplers (Kodak 1949)

Ferraniacolor—Monopack negative-positive film
Filmcolor—Autochrome mosaic screen on film (ca. 1933)
Finlay—Separable regular mosaic-screen plate (**Finlaychrome** was the corresponding inseparable screen plate)—see Paget (below)
Flexichrome—Hand-colouring process on relief positives (Kodak 1950)
Francita—Three-colour additive cinematograph process using beam-splitting device

Gasparcolor—Three-colour monopack cinematograph process, using the dye-bleach process (1934)
Gaumont—Three-colour additive cinematograph process, using triple-lens system (1912)
Gevacolor—Monopack films, reversal and negative-positive types

Harriscolor—Two-colour subtractive cinematograph process (1929)
Hérault Trichrome—Three-colour cinematograph process (ca. 1929)
Horst—Three-colour additive cinematograph process

Ilford Colour—Subtractive monopack reversal film (1948)
Irix—Three-colour imbibition printing process (1935)
Isocolor—Three-colour transfer print process (U.S.A. ca. 1942)

Johnson Colour Screen Process—Separable mosaic-screen process, on Finlay principle (1951)
Jos-Pé—Three-colour imbibition process using tanning development (1926)

Keller Dorian—Lenticular cinematograph process (1925)
Kinemacolor—Two-colour additive cinematograph process using rotary colour shutters (1906)
Kodachrome*—1. Two-colour transfer printing process (1915)
2. Three-colour reversal monopack film (1935)
Kodacolor*—1. Lenticular cinematograph process for 16-mm. film (1928)
2. Three-colour monopack negative film and positive paper process for amateur use (1942)
Kotavochrome—Colour prints on white cellulose acetate base from large-size Kodachrome transparencies (1941)
Krayn—Mosaic-screen cinematograph film (ca. 1907)

Leto—An early screen plate
Lignose—Irregular mosaic-screen film (1927)
Lumicolor—Autochrome screen on film (ca. 1933)

Magnachrome—Two-colour additive cinematograph process
Magnacolor—Two-colour subtractive cinematograph process (1930)
Minicolor—Colour prints on white cellulose acetate base from 35-mm. Kodachrome transparencies (Kodak 1941)
Morgana—Two-colour additive cinematograph process (1932)
Multichrome—Negative–positive monopack film. Similar process to Agfacolor (Hansard Photographic Laboratories Ltd. 1952)
Multicolor—Two-colour bipack subtractive cinematograph process (1929)

Neotone—Imbibition printing process

Omnicolor—Mosaic-screen plate (1907–1914)
Opticolor—Lenticular cinematograph process
Ozobrome and Ozotype—Printing processes resembling Carbro (1898)

Paget—Regular mosaic-screen separable plate (1912) (known since 1929 as the Finlay plate)
Pakolor—Negative-positive monopack film, similar to Agfacolor (Photo Chemical Co. Ltd. 1952)
Pan Chroma—Relief film, transfer print process (U.S.A.)
Pantachrome—Lenticular film with multilayer emulsion (Agfa)
Pavelle—Three-colour monopack prints on Agfa–Ansco Printon material for reproducing colour transparencies
Photocolor—Two-colour subtractive cinematograph process using twin-lens camera (ca. 1930)
Pilney—Two-colour subtractive cinematograph process (1930)
Pinachrome—Three-colour printing process using leuco dye bases which oxidise on exposure to light
Pinatype—Three-colour imbibition printing process (1905)
Plenacolor—Negative-positive monopack process (Agfa–Ansco Corpn.)
Polacolor—Cinematograph process (Polaroid Corpn. of America 1947)
Polychromide—Chemical toning process
du Pont S. and T. Film—Cinematograph monopack film for printing from Technicolor negatives; emulsions dispersed in resinous colour-coupler (1949)
Printon—Monopack printing material (Agfa–Ansco Corpn.)

Raycol—Two-colour additive cinematograph process (1930)
Raydex—Early form of three-colour Carbro
Raylo—Printing process resembling Carbro (ca. 1923)
Rouxcolor—Three-colour additive cinematograph process using beam-splitting device (1948)

Sennetcolor—Subtractive cinematograph process (1930)
Sirius—Two-colour subtractive cinematograph process (1929)

* See note at end of Appendix A.

Spectracolor—Two-colour subtractive cinematograph process

Splendicolor—Three-colour subtractive cinematograph process (1928)

Technicolor—Subtractive cinematograph process—two-colour (1915); three-colour (1933)

Thames—Early name for Finlay screen process

Thomascolor—Three-colour additive cinematograph process (U.S.A.)

Triadochrome—Chemical toning process (for three-colour prints) (ca. 1920)

Trucolor—Two-colour subtractive cinematograph process

Ufacolor—Two-colour subtractive cinematograph print process using inorganic toning (ca. 1937)

Utocolor—Three-colour printing process using a bleach-out principle (Paris ca. 1911)

Uvachrome—Dye toning process based on copper ferrocyanide (ca. 1916)

Versicolor—An early screen plate

Vitacolor—Two-color additive cinematographic process (1930)

Vivex—Three-colour printing process resembling Carbro (1932)

Warner-Powrie—Regular mosaic-screen plate (1905) (not issued commercially)

Wash-off Relief—Three-colour imbibition print process (Kodak 1936)

Zoechrome—Three-colour subtractive cinematograph process

* **Nomenclature of Kodak Colour Materials**—The names given to Kodak colour materials at present have the following significance—The prefix *Koda-* is used for materials processed by Kodak, and *Ekta-* for materials that may be processed by the user. The suffix *-chrome* is used for reversal materials, and *-color* for non-reversal materials, negative or positive.

B—Bibliography and Literature Review

The field is well covered by a few standard text-books. Thus, for the historical development of the subject as well as theoretical and technical discussion, the works by Friedman [51] and Wall [153] may be consulted, and also Klein's monograph [81]†, which has special reference to cinematography. Wall's book in particular is a mine of information on older processes; it has reference to about 150 books, 3000 patents, and the work of about 2000 authors. Unfortunately it is out of print and difficult of access. Spencer's volume [128] is a recent work dealing more particularly with the practical operation of current processes, and is lavishly illustrated in colour. A standard work on photography e.g. [105, 111] may be referred to for details of developing processes, etc.

General descriptions of modern processes, diagrammatically illustrated in colour, are given by Mees [106] and in the manufacturers' (non-technical) publications e.g. [33, 41, 92]. The following papers also include material descriptive of commercial processes, and some historical data [9,10,64, 66,109,104,125,147,156], and much useful information is given in the FIAT report by Schneider [134].

Complete details of the manufacture and principles of operation of the Agfacolor films have been given in a series of reports on German industry, of which the principal ones are those by Adams [1], Chilton [21], Ranger *et al.*[120], and Schneider [138]. Keck's report [77] in the same series describes the colorimetric principles of colour photography and their particular application to the Agfacolor films.

The files of the *British Journal of Photography* and the *Photographic Journal* are also a valuable source of information.

A number of recent books deal with colour vision, e.g. those by Hartridge [62], Willmer [158], and Wright [159]. These are comprehensive treatises; for more popular, but authoritative accounts, see Hartridge [61,63] and Mann and Pirie [99]. For works on colorimetry, see e.g. Bouma [14], Evans [47], Wright [157], and a less technical account given by Chamberlin [20].

† Since this review was written, a third and expanded edition of this work has appeared [81].

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ERRATUM

Quantitative Relationship between Depth of Dyeing and Light Fastness. J. C. Eaton, C. H. Giles, and M. Gordon (*J.S.D.C.*, **68**, 395 (Oct. 1952))— The abscissæ in Fig. 1 are, of course, **Depth,** $\%_{o}$, not "log (Depth, $\%_{o}$)".

Notes

Proceedings of the Council

At a meeting of the Council held at the offices of the Society, 19 Piccadilly, Bradford, on 23rd July 1952, the proceedings included the following items of interest—

LONDON LECTURE— It was resolved after full discussion that Council should give its support to the proposal of the London Section Committee to hold an annual London Lecture.

I. S. O. CONFERENCE IN AMERICA — It was reported that arrangements had been made for the Society to be represented at the third meeting of ISO/TC 38/SC1— Colour Fastness of Textiles, to be held in New York on 10–11th November 1952, by Dr. P. W. Cunliffe (Chairman of the Fastness Tests Co-ordinating Committee) and Mr. E. Wilson (a member of the F.T.C.C.). The Society was greatly indebted to the respective employers, the British Cotton & Wool Dyers' Association Ltd. and the Bradford Dyers' Association Ltd., and to the President for enabling these arrangements to be made.

DIPLOMA COMMITTEE— It was resolved to invite Dr. M. Crawford of the Belfast College of Technology to serve on this Committee.

B.S.I. SILK INDUSTRY COMMITTEE— It was resolved to invite Mr. E. G. Harris to represent the Society on this Committee.

YORKSHIRE COUNCIL FOR FURTHER EDUCATION — It was resolved to invite Mr. J. G. Evans to represent the Society on the County Advisory Committee for Chemistry.

ROYAL TECHNICAL COLLEGE, SALFORD— The resignation of Mr. F. Farrington was accepted with regret and with thanks for his past services, and it was decided to invite Mr. J. W. Reidy to represent the Society on the Applied Science Advisory Committee.

MEMBERSHIP— Eleven applications for ordinary membership were approved.

Meetings of Council and Committees October

Council— 8th
Finance— 8th
Publications— 21st
Colour Index Editorial Panel— 22nd
Review of Textile Progress— 16th
Fastness Tests Co-ordinating— 20th
Printing Symposium— 16th
Historical Records— 3rd
Abstractors— 31st

Deaths

We regret to report the loss by death of Mr. H. Barnes and Mr. F. Wilson.

Award of Olney Medal

The Olney Medal of the A.A.T.C.C. has been awarded for 1952 to Werner von Bergen, a pioneer of wool research in America and co-author of the *American Wool Handbook*.

Finance of Chemical Publications

According to *Chemistry and Industry* for 30th August 1952 (p. 853), the British chemical publishing societies and the Bureau of Abstracts have been faced with a deficit of £50,000 for 1952. As it would be a serious blow to the scientific prestige of this country if the Bureau and the societies had to close down their publishing activities, Messrs. Imperial Chemical Industries Ltd. have at once made a special contribution of £25,000 in the expectation that other firms in the chemical and allied industries will contribute the balance of the deficit.

British Chemical and Dyestuffs Traders' Association

The new address of the Association is 12 York Buildings, Adelphi, London W.C.2 (telephone WHitehall 4963–4).

Verband Schweizerischer Kleiderfärbereien und Chemischer Reinigungsanstalten

The Annual Conference was held on 15–16th June 1952 in Kandersteg, and was attended by the presidents of the French and German associations.

Verein deutscher Färber

A new local section, in Hessen, was formed at a meeting held in Fulda on 24th May 1952, at which over forty members were enrolled. Meetings are to be held every other month.

Indian Jute Mills Association Research Institute

New buildings in Calcutta, formally opened by Mr. Nehru on 2nd January 1952, include laboratories for dyeing, finishing, and chemical technology. The new premises provide modern amenities for a staff of 85, including 45 qualified scientific men or technologists.

Science and Technology Teachers

On 3rd July 1952 Mr. Boyd-Carpenter stated in the House of Commons that of the graduates teaching full time in grant-aided establishments of higher education in England and Wales in 1951, 1444 had degrees in mathematics or natural science and 776 in engineering or technology. In the universities of Great Britain there are approx. 3100 teachers of science and technology.

Surface-active Agents in America

Total (1951) U.S. output of surface-active agents was 683 million lb., an increase of 1% on 1950. Sales in 1951 amounted to 587 million lb. valued at $125 million. Anion-active agents accounted for nearly 90% of total production in 1951. Production of cyclic agents increased by 20%, while that of acyclic agents fell by 23% in 1951 compared with 1950.

Fullers' Earth

It is recommended by the Fullers' Earth Union Ltd. that the term *fullers' earth* should be generally reserved for adsorbent clays composed mainly of calcium montmorillonite.

Lithium Hypochlorite as Bleaching Agent

Laboratory tests in the U.S.A. have shown this compound to be a completely soluble, stable, and highly active bleaching agent, suitable for use in the home, and large-scale pilot-plant production is planned.

Spanish Phenol, Polyvinyl Chloride, and Titania

It is reported that the Union Química del Norte de España (the so-called "Spanish little I.G.") is now making 5 tons of phenol and 8 tons of formaldehyde a day. This autumn manufacture is to be begun, for the first time in Spain, of titanium dioxide pigment from Spanish ilmenite. The Electroquímica de Hernani is making polyvinyl chloride (2 tons/day), part of which is used for fibre production in Bilbao.

Sulphonation with Sulphur Trioxide

A plant being built in Chicago for the manufacture of sulphonated hydrocarbon detergents and surface-active agents is to employ stabilised sulphur trioxide as the sulphonating agent.

Prediction of the Colour of Organic Dyes from their Chemical Formulæ

At a joint meeting of the Society of Chemical Industry and the Basle Chemical Society on 20th May 1952, Professor W. Kuhn showed how a one-dimensional free-electron gas model can be used to explain the light absorption of symmetrical polymethins, porphyrins, and phthalocyanines. For polyenes, including the carotenoids, the π-electrons are no longer free but are subjected to a potential of one-dimensional sinusoidal periodicity. The lecture was illustrated by many ingenious models, diagrams, and slides.

Chlorophyll Production

The chief use of water-soluble chlorophyll derivatives, now generally sodium copper-chlorophyllins, has been in wick-type room deodorants and as colouring or masking agents in soaps, but incorporation in personal deodorant preparations is expected to increase U.S.A. sales from $22 million in 1951 to $50 million in 1952. Four companies are now producing chlorophyll, mainly by solvent extraction from alfalfa, a ton of which yields 4–8 lb. of chlorophyllin selling at $85 per lb.[1]. One American firm purchases its chlorophyll supplies from Great Britain.

[1] Arthur D. Little Inc. (Cambridge, Mass.), *Industrial Bulletin* (June 1952): *Chem. Trade J.*, 131, 225 (25th June 1952).

World Production of Man-made Fibres in 1951

The *Textile Organon* estimates world production (excluding that of the Soviet bloc and China) to be (in million lb.) 4218, representing an increase of 15% on the 1950 figure. Of the 1951 total, rayon and acetate rayon accounted for 3957. The world production capacity is estimated to be 5174.

Grilon Polyamide Fibre

Grilon, an all-Swiss synthetic fibre based on caprolactam, is now being synthesised by the Holzverzuckerungs-AG. at Domat-Ems and spun by Fibron S.A., a subsidiary company.

Perlon Trade Mark Association

The PERLON-Warenzeichenverband e.V. has been formed, with headquarters at Frankfurt-am-Main, by the six manufacturing firms— Basf, FBy, Kunstseidefabrik Bobingen, Rottweiler Kunstseidefabrik AG., Deutsche Rhodiaceta AG., and Vereinigte Glanzstoff-Fabriken AG.— and the Gesamtverband der Textilindustrie in der Bundesrepublik Deutschland.

Fiberfrax — a Vitreous Aluminium Silicate Fibre

Fiberfrax, made by the Carborundum Co., Niagara Falls, N.Y., U.S.A., is a vitreous

aluminium silicate fibre, which can be heated to 2300°F. without loss of properties and does not soften even at 3000°F. It is made by fusing alumina and sand at 3300°F. and treating the molten material with a controlled blast of air to produce a fluffy mass of extremely fine, randomly arranged fibres up to 3 in. long and of average diameter 4 μ. C O C

Research Council of Israel

According to the Annual Report for 1950–51 (abstracted in *Nature*, **170**, 69 (12th July 1952) the Textile Research Association has continued work on the use of acid bromates for rendering wool shrink-resistant. A survey of the source of raw materials for the manufacture of cellulose has been undertaken, and a study made of micro-organisms responsible for the retting of flax.

Work on Silk Dyeing

It is reported that Section VIII (Dyeing) of the International Silk Association is studying the possibility of wash-fast dyes in brown and navy blue. The Technology and Research Committee is studying technical suggestions concerned with the degradation of silk under fast-dye treatment, detection of lousy silk, photodegradation of silk, and risk of damage from scrooping.

Combustion Products of Plastics

According to the report for 1951 of the Fire Research Board of the D.S.I.R.[1], the principal gaseous products of combustion of chlorinated methacrylate resins and polyvinyl chloride plastics are carbon dioxide and carbon monoxide with some hydrogen chloride, but only traces of carbonyl chloride (phosgene) are found.

[1] H.M.S.O. (2s. 0d.); *Chem. Trade J.*, **131**, 488 (29th Aug. 1952).

U.S. Patent Law Revision

One of the main changes resulting from a major revision and codification of the patent laws, to come into effect on 1st January 1953, is that it will be possible to receive a patent for a new use of an old product.

Non-aqueous Titration of Very Weak Acids and Bases

According to *Industrial and Engineering Chemistry* for August 1952 (p. 20A), phenols may be rapidly determined, without interference from amines, aromatic hydrocarbons, or small amounts of alcohols, by titration with sodium methoxide in benzene–methanol using a visual indicator. *o*-Nitroaniline in ethylenediamine or dimethylformamide may be used for determining weakly acidic substituted phenols. The accuracy of phenol determination is 0·2–0·5%, and it may prove possible to differentiate between certain carboxylic acids and phenols, as well as between aromatic and aliphatic amines.

Circulation of Scientific Information

Among recommendations made by a deputation from the Parliamentary and Scientific Committee that waited upon the Lord President of the Council on 28th July 1952 was that urgent action should be taken in regard to increasing facilities in this country for printing and circulating scientific information.

Locating Information

The Intelligence Division of D.S.I.R. recently undertook the publication of a bulletin, *Unanswered Questions*, in which answers were invited to readers' questions when normal sources of information had apparently failed. Although great interest was aroused, the expected flood of questions did not arrive.

In order further to test the efficacy of abstracting services, a search for readers' actual answers to "unanswered questions" was made in existing abstracts. A junior member of the staff, with little previous experience, used author indexes and found 84% of the references. Two more experienced searchers, using subject indexes and guides, found fewer than 50% of the abstracts which existed.

The conclusions drawn are that probably 90% of useful scientific and technical papers are eventually abstracted, but that the main problem is to find the abstracts.

Codification of Organic Compounds

At its meeting in New York on 10th September 1951, the Commission on Codification of the International Union of Pure and Applied Chemistry concluded that the Dyson notation (cf. J.S.D.C., **63**, 270 (1947); **65**, 541 (1949)) should be adopted as the provisional international system. It recommended also that particular attention should be given to the inclusion, whenever possible, of the desirable features of all other notations, especially those of Gruber and Wiswesser. The Commission considers it essential that the notation finally accepted shall be capable of generating a system of nomenclature in harmony, as far as possible, with current practice.

Chemical-formula Typewriter

It is reported in *Chemical and Engineering News* (23rd June 1952) that a typewriter has been specially constructed in the U.S.A. for the direct typing of ring structures and other complex chemical formulæ.

OBITUARY NOTICES

Charles Édouard Philemon Marie Callebaut

It is with very deep regret that we record the death of Mr. C. É. P. M. Callebaut at the age of 72. Born at Alost in Belgium, he graduated in Civil Engineering at Ghent University at the age of 21. He first joined a firm of boiler surveyors as an inspector, but after five years he left to join Gustave de Keukelaer of Brussels in the manufacture of

dyeing machinery. Here he was joined by a young chemist, Julien de Blicquy, and so began a close association which lasted over forty years.

Before the First World War Callebaut left Gustave de Keukelaer to start in business for himself in Brussels, but when the Germans overran Belgium his business was forcibly closed down and his plant taken by the Germans, and so in 1915 he came to England and became engaged in the manufacture of armaments.

In 1919 he joined with other business associates to form the Longclose Engineering Co. Ltd., and at his death he was Chairman and Managing Director. In 1937 he became a naturalised British subject.

Charles Callebaut had a dynamic personality, and was admired by all who knew him for his very great courage and generosity. He had a passion for detail and possessed a most inventive and ingenious brain, which was largely responsible for his success.

He was a member of the Society for some twenty years.

To his widow and only daughter we extend our very deep sympathy. F. H. MARSH

Gustav Ullmann

Members of the Society, especially members of the Manchester Section, will miss the genial presence at Society meetings of Dr. G. Ullmann, who died in Manchester after a short illness on 7th April 1952, when he was in his 77th year. Dr. Ullmann was one of the few remaining representatives of the old and well established school of Continental textile colourists. The son of a physician at Znaim in Austria, he was educated at the local *Gymnasium* and studied chemistry at the Universities of Vienna and Basle. He was for some time assistant to Professor P. Friedländer at the Technische Hochschule in Vienna. On the completion of his academic studies, he specialised in textile technology; he gained practical experience by working at various Austrian spinning, weaving, and dyeing mills, and was later associated with the Niederlahnstein Maschinenfabrik AG., where he gained first-hand knowledge of the construction and mechanisms of textile machinery.

Early in the century he published *Die Apparatefärberei*, perhaps the first textbook on pack-systems in bleaching and dyeing. He had later many inventions to his credit, notably those relating to kier-boiling and to water treatment.

From 1904 until the First World War he was an executive director of the Vereinigte Osterreichische Textilindustrie AG., at that time an important concern. After the war Dr. Ullmann established his own firm "Lia" in Vienna for planning textile mills. He worked as an independent consultant, and his advice and counsel were sought and appreciated by many persons and firms. In this kind of work he found pleasure and satisfaction. He was always ready to offer advice and assistance, and his great knowledge and experience soon made his firm well known on the Continent and overseas. He was one of the founder members of the Internationaler Verein der Chemiker-Koloristen, attended its yearly conferences, and through it expanded the wide circle of his friends. Unfortunately, political events and racial persecution forced him to terminate his activities in Vienna in 1938 and made it impossible for him to remain in Austria. He came to this country in the early months of 1939, and settled in Manchester. He soon made contact with textile mills, and he continued in the profession of textile and industrial consultant.

His interests extended into the field of pulp and paper-making, and he was for many years connected with the East Lancashire Paper Mill Co. Ltd., Radcliffe, where, in conjunction with Dr. P. Bean, he studied the use of cereal straws for paper-making and for animal feeding stuffs. He described some of his work and ideas in an article on "Making Animal Feeding Stuffs in Bulk" in the *Times Review of Industry* (July 1950).

In conjunction with Dr. Bean, he contributed a paper on "Some Relations, Economic and Technical, between the Pulp and Paper Trade and the Textile Trade", which was read to the Manchester Section of the Society (J.S.D.C., **66**, 576 (Nov. 1950)). Elsewhere he wrote amusingly on the technique of sleeping at lectures.

Dr. Ullmann was a member of the Manchester Section, and he rarely missed any of its lectures and other functions. To the end of his life he maintained a keen interest in developments and inventions relating particularly to textiles; he was very well informed regarding the literature of his profession, and was never happier than when sharing his knowledge and experience with others. He will long be remembered by his friends for his fine qualities of kindness, generosity, and unselfishness, and for his quiet humour; the Society has lost an active, loyal, and distinguished member.

A. F. KERTESS
F. SCHOLEFIELD

New Books and Publications

Voprosy anilinokrasochnoy khimii
Trudy VIII soveshchaniya po anilinokrasochnoy khimii i tekhnike
[Questions of Aniline Dye Chemistry]
[Transactions of the Eighth Conference on Aniline Dye Chemistry and Technology]

Pp. 181. Moscow and Leningrad: Izdatelstvo Akademii Nauk S.S.S.R. 1950. [In Russian.] Price, 8.50 roubles.

This volume contains the more important papers presented at a conference on dye chemistry held in December 1947. This and the seven previous conferences—the first was in 1937—were organised by the late A. E. Poray-Koshits.

The first three papers consist of appreciations and reminiscences of the organic chemist N. M. Kizhner (1867–1935), who, with the coming of the Soviet régime, devoted himself to the development

of the dye-manufacturing industry. The next paper is again an appreciation of a worker who was prominent in the development of Soviet dye-making— M. A. Ilyinsky (1856–1942), a pioneer in anthraquinone chemistry and a prominent industrial research worker in dye chemistry in Russia and in Germany during the period before the First World War. Ilyinsky discovered (1891) that, in presence of mercury salts, anthraquinone sulphonates in the *a*-position, and he postulated the intermediate formation of a compound containing the group -Hg·SO_4H in this position. This postulate has been confirmed by Kozlov, who gives a review of his own work on the formation abd hydrolysis of anthraquinonesulphonic acids in one of the papers in the collection. The remaining nine papers are to be found in abstract in this issue of the *Journal* (pp. 464–466, 468 and 477).

One of these papers, by Blinov on the development of Variamine Blue, directly concerns the technology of dyeing. It contains a number of obscurities and is on a lower scientific level than the remainder of the collection. Two papers deal with the diazotisation reaction. One of these, by Vernikovskaya, is of value as a collection of data, though the basis of her interpretation of these data can no longer be maintained, and was disputed at the time by B. A. (son of A. E.) Poray-Koshits in the second of these papers.

Of the remaining articles, the paper by Dokunikhin on vat dyes deserves special mention. One of the participants in the discussion on the paper, who was very adverse in his criticism of some of Dokunikhin's conclusions, said that the amount of material in the paper was great, but its treatment superficial. There is some truth in this assertion. Nevertheless, the reviewer found the mass of factual material in this paper of great interest, especially the very lucid discussion of the various leuco esters derived from indanthrone.

A. E. Stubbs

Förädlad Textil
[Finished Textiles]

By T. Ahlstrand and T. Vinell. Pp. 175. Sandared, Sweden: AB. Alf Stigens Fabriker [1952]. [In Swedish.] No price.

In Scandinavia it is seldom that a birthday or anniversary is allowed to pass without some form of celebration. For companies, it is not uncommon to mark an important occasion by the publication of a book. These books are usually of high standard, judged from any aspect, and *Förädlad Textil*, issued to mark the twentieth anniversary of the founding of AB. Alf Stigens Fabriker, is no exception.

The common form is a factual account of the firm's progress, tinged with self-eulogy however modestly disguised. This is where the book under review differs from others, for over half (pp. 9–99) is devoted to an outline of the history of the art of dyeing, from the earliest recorded annals. The authors have left no stone unturned in their search for truth, as is shown by the list of 45 references. The various natural dyestuffs in use in ancient times are described, with methods of extraction from plants, insects, and crustaceans. Drawings, tables, and photographs show the sources of these dyestuffs.

Every country in which the art of dyeing was practised receives its due recognition, and the developments and influences which provided stimuli to progress are unfolded. Evidence from China, India, Egypt, Phœnicia, Persia, Greece, etc. is presented, showing that the tinctorial arts were established as far back as 2500 B.C. The contributions of Holland, Italy, France, Germany, and England, up to the time of W. H. Perkin, are given appropriate recognition. It is shown that, with a limited range of dyes, primitive equipment, and little more than empirical knowledge, ingenuity had great scope, and the good dyers inevitably emerged from the rut and became famous.

Between the fifteenth and the seventeenth centuries, dyers received recognition from kings and governments. Guilds were sanctioned in several European countries. The élite were thereby segregated and distinguished by the use of guild signs and seals, many of which are beautifully illustrated in colour opposite p. 32.

Chapter II contains a parallel story of the art of dyeing in Sweden. Ancient records, prints, tapestries, and the oldest dyers' pattern cards are depicted. The reader is taken back to the days when the development of new dyes was dependent upon the botanist and naturalist. The university town of Lund was the centre of learning, and the Professor of Natural History made a special study of plant life in search of new dyes. The Swedish literature of dyeing in olden days is reviewed, e.g. that of Johann Linder (1720), Erik Salander (1727), Carl von Linné (Linnæus) (1742). Information on the development and location of the industry is given in this section.

Chapter III could well be entitled "The Stigen Story"— it is a fascinating account of a young man's training, the benefit he derived from it, his courage, and his enterprise. His creation is a shining example of reward for hard work. What was a very unpretentious affair in 1932 becomes a fine modern factory, with splendid equipment for dyeing, finishing, printing, and the manufacture of leathercloth.

There is much information about machines, and a useful table showing which classes of dyes may be used for every fibre in common use. Novel finishes are described and illustrated. Modern trends in dyeing are discussed up to the Standfast molten-metal and the American hot-oil processes. Examples of photoprints are shown in colour photographs.

The final chapter, entitled "Wider Possibilities with New Fibres", is a summary of regenerated and synthetic fibres, with notes on the manufacture of the last named group and on their properties. Nylon, Perlon, Rhovyl, Orlon, Terylene, Ardil are the subject of speculation as regards new uses.

The photographic work is excellent and the book itself is bound in white Stigolon leathercloth with gold lettering, making a valuable addition to the bookshelf of a dyer or a colourist.

H. Boothroyd

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.)*

I—PLANT; MACHINERY; BUILDINGS

Smouldering of Dusts. F. E. T. Kingman and K. N. Palmer. *Chem. and Ind.*, 739 (26 July 1952).

A brief preliminary report on the minimum depths (0·1–3·0 cm.) of different dusts necessary for smouldering to be sustained. J. W. B.

Protection of Printing-machine Boxwheels. F. Schwenk. *Dyer*, **108**, 243–245 (22 Aug. 1952).

Protection against accidents in operating printing machinery due to exposed boxwheels is effected by fixing a pair of semicircular doors, in which are cut apertures corresponding to the number of rollers in the printing machine and to their normal position when mounted. The diameter of these apertures is a little larger than that of the boxwheel. On each boxwheel is fixed, by a very simple device, a round plate or disc of sheet-iron slightly larger in diameter than the aperture in the door, and revolving with metal-to-metal contact on the greased inner side of the door. B. K.

Comparative Drying Rates—Infrared *versus* Hot Air. R. E. Wingard and W. H. Rozier. *Alabama Polytech. Inst. Bull.*, **47**, (4), *Eng. Expt. Sta., Eng. Bull.*, (15), 3–14 (1952): *Chem. Abs.*, **46**, 6870 (10 Aug. 1952).

In a modified compartment-type drier a combination of steam and infrared heating gave with cotton cloth the highest rates of drying and the lowest drying times. Steam heating alone gave the lowest rates and the longest times. Infrared heating alone gave more rapid drying than steam, but the rates were not quite as high as for combined steam and infrared heating. C. O. C.

PATENTS

Coating Rollers and Cylinders with Thermoplastics. Z. Lorentzen. *BP* 678,367

Combustible Mixture for Singeing Machines. Sir James Farmer Norton & Co. *BP* 678,180

Apparatus for using either liquid fuel or gas. C. O. C.

Package Dyeing Machine. Courtaulds. *BP* 677,472

A single mixing chamber is connected to several units. Each unit consists of a tank, a pump, and controllable outflow and inflow channels arranged so that each unit can be used invidually and independently by circulating liquor from the tank through the packages in the tank. Any number of units up to the total present may be used collectively by continuously withdrawing liquor from their tanks to the mixing chamber and directing back to them the mixed liquor from the mixing chamber. C. O. C.

Package Dyeing Machine. Obermaier & Cie. *BP* 678,251

A pipe elbow having a horizontal part to receive adjacent to its free end a propeller for feeding the dye liquor and an upright part whose upper end forms a seat for an exchangeable hollow support for the goods to be dyed is placed in the dye vat. This support also serves to separate the dye vat from the heating and mixing chamber for the dye liquor. C. O. C.

Hank Drying. H. Fleissner & Sohn Maschinenfabrik. *BP* 678,252

If the bars which carry the hanks through the drying chamber turn counter to the adjacent carrier bars, so that the adjacent layers of threads of the hanks on two adjacent carrier bars move in the same direction, all danger of entanglement is eliminated. C. O. C.

Drying Warps, etc. in Open Width. Algemene Kunstzijde Unie. *BP* 677,756

A drier which does not flatten the threads and in which the heat is most economically employed. C. O. C.

Untwisting Material in Twisted Rope Form. Bradford Dyers' Assocn. *BP* 677,647

A simple device for continuously taking the twist out of roped material, e.g. pieces coming from a hydroextractor. C. O. C.

Scrimping Bars. J. Dungler. *BP* 677,102

The bar comprises an axially curved spindle, several rollers independently rotatable on the spindle, and a flexible sleeve surrounding the rollers. Each roller has a central bore the axial cross-section of which is convex with respect to the spindle, to allow the rollers to assume inclinations at varying angles. This reduces inertia of the moving parts to a small value and reduces the friction between them. C. O. C.

Bobbins of Cloth Expanders. F. Smith & Co. (Whitworth). *BP* 677,555

A bobbin bearing having improved lubrication. C. O. C.

Feeding Webs between a Series of Rollers. Pembertons (Gateshead). *BP* 677,331

Feeding of fabric, e.g. through a calender, is effected, without need to guide by hand the ends of the fabric between adjacent rollers, by using a series of endless-belt units each consisting of an endless belt arranged about two pulleys deriving their motion from the rollers. Each belt unit is placed on and pressed against the fabric-carrying side of a roller so that the inner pulley of the first or leading unit and both pulleys of the other units are between adjacent rollers. C. O. C.

Handling Tubular Fabrics. Samcoe Holding Corpn. *USP* 2,590,938

Apparatus for forwarding the fabric without imparting longitudinal tension. C. O. C.

Batching and Drying Tubular Fabric. I. Pasold. *BP* 678,055 *BP* 678,328

Preshrinking and Setting Tubular Textile Fabric. Samcoe Holding Corpn. *USP* 2,589,344–5

Apparatus is described in which the fabric is longitudinally compressed before, during, or after steaming, so as to render it dimensionally stable when washed. C. O. C.

Effecting Liquid Penetration of Fabrics. R. D. L. Frederick. *BP* 676,781

The fabric is drawn continuously through liquid in a vat, especially that of a jig, the liquid being continuously sucked through the submerged fabric first in one direction and then in the other direction. C. O. C.

Cylinder Drying Machine. F. Sibel. *USP* 2,591,307

An economical, easily controlled cylinder drier is described which is inherently quick to respond to temperature change requirements; thus if movement of the fabric is stopped, the cylinder cools so quickly upon the heat being turned off that no damage is caused to the fabric remaining in contact with the cylinder. C. O. C.

Tenter. Proctor & Schwartz. *USP* 2,591,621

A tenter which can be worked at high speeds has the air nozzles arranged above and below the path of the fabric, so as to discharge large volumes of drying air against both sides of the fabric without tending to disengage it from the tenter pins. The fabric is kept relaxed throughout drying. C. O. C.

Applying Dry Steam simultaneously to Both Sides of a Fabric. Samcoe Holding Corpn. *BP* 676,166

A device whereby steam can be applied to both the upper and lower sides of fabric, and condensed water is prevented from reaching the fabric. C. O. C.

Preparing Printing Screens. J. C. Pownall and A. R. Dale. *BP* 677,630

The screen is mounted on top of a frame. A suction head is mounted beneath the frame so that it can be applied to any part of the undersurface of the screen to suck the printing colour through the open parts of the screen and retain it for further use. The edges of the design remain sharp and clear, not rough and irregular as when the colour is removed by rubbing with rags. C. O. C.

Presses for Knitted Garments. Brown & Green. *BP* 678,602

Cleaning Emergent Vapours from Dry-cleaning Plants. R. Decker and H. Holz. *BP* 677,700

Gases from textile machinery, e.g. hot-air drying vapours from dry-cleaning plant, which are contaminated with dust, fluff, etc., are passed into a chamber wherein a liquid screen is formed by multiple spray-nozzles. Impurities are removed, fall to the bottom of the container, and are drawn off through a liquid seal; cleaned gas is discharged at the top. J. W. B.

Lytax Textiloscope (IX p. 478).

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Acetylene in Chemical Industry. N. Booth. *Chem. and Ind.*, 812–819 (23 Aug. 1952).

A survey of the production, handling, and, in particular, use in syntheses, of acetylene. J. W. B.

Structure of Lanostenol. R. G. Curtis, J. Fridrichsons, and A. M. Mathieson. *Nature*, **170**, 321–322 (23 Aug. 1952).

The structural formula for lanostenol is elucidated from Fourier synthesis of the iodoacetate; it is shown that the formula cannot be fitted to the "isoprene" rule which normally applies to triterpenes. As lanostenol coexists with cholesterol in wool wax and with ergosterol in yeast, the question arises as to whether it should be considered a triterpene or a trimethylated sterol. J. W. B.

Iron-containing Oleines. M. Kehren. *Melliand Textilber.*, **33**, 853–855 (Sept. 1952).

Iron present in textile oils containing fatty acids is readily removed from the fabric in a cold alkaline scour and cannot give rise to the yellowish streaks and flecks that have been attributed to it. It has no significant effect on the autoxidation of the oil, but its presence causes errors in the determination of the thiocyanogen value (T.V.) of the oil, which is of importance in the determination of the concn. (= I.V. − T.V.) of highly unsaturated acids, which are prone to autoxidation. A revised method of determining the T.V. is described. A. E. S.

Consumption of Organic Synthetic Detergents in the U.S.A. C. P. Neidig and A. B. Hersberger. *Chem. Eng. News*, **30**, 3610–3615 (1 Sept. 1952).

In 1939 the greater part of the synthetic detergents was used by the textile industries, but now such use is only about 7% of the production. In 1951 37% of all detergents production was synthetic. C. O. C.

Studies in the Surface Tension Measurement of some Colloidal Solutions. I—Gum Arabic, Gum Tragacanth, and Dextrin Sols. S. N. Banerji. *J. Indian Chem. Soc.*, **29**, 270–274 (April 1952).

Determination of the surface tension of gum arabic, gum tragacanth, and dextrin sols at various concentrations and temperatures has been carried out using a modified form of Traube's stalagmometer. The surface tension of gum arabic sol decreases progressively with increase in the concentration of acid added, whereas the addition of alkali increases the surface tension in a similar manner. The surface tension of gum arabic is also lowered by the addition of metallic salts, the order of the effect being $Li > Na > K > Ca > Ba$. These observations are explained on the basis that reversible colloids contain some dissolved material in true solution under the following conditions of equilibrium—

Simple molecules ⇌ Aggregated molecules ⇌

Colloidal particles

C. H. R.

PATENTS

Emulsifying Agents. General Aniline. *USP* 2,593,112

The addition products of 10–25 mol. of ethylene oxide and dialkylated phenols (Alk of > 5 C; total C > 14) are extremely efficient agents for preparing aqueous emulsions of oils. C. O. C.

Aqueous Soapless Detergents. ICI. *BP* 678,445

A stable aqueous detergent consists of 8% of an anion-active detergent and a sodium salt of a carboxymethyl cellulose having 0·7–0·8 sodium carboxymethyl group per anhydroglucose unit of cellulose and still containing the NaCl, Na_2CO_3, and sodium glycollate associated with its manufacture. C. O. C.

Capillary-active Derivatives of Benzenesulphonamides. Henkel & Cie. *BP* 678,004

Washing, wetting, emulsifying, etc. agents are obtained by treating with ethylene oxide a benzenesulphonamide containing at least one reactive H atom on the N atom and an alkyl or *cyclo*alkyl group of > 3 C in the nucleus. C. O. C.

Fatty Quaternary Acylamidoalkylammonium Ester Salts—Antistatic and Softening Agents. Lankro Chemicals. *BP* 678,604

Compounds of formula—

$$R^1\text{·CO·NR}^2\text{·CH}_2\text{·CHR}^3\text{·NR}^4\text{R}^5\text{·R}^6\text{·O·CO·R}^7\}X$$

(R^1 = sat. or unsat. Alk or aralkyl of 8–20 C; R^2 = H or subst. or unsubst. Alk or aralkyl; R^3 = H or Alk; R^4 and R^5 = H, Alk, or hydroxyalkyl; R^6 = alkylene; R^7 = Alk of 11–17 C; X = Br, Cl, or I), e.g.—

$$C_{17}H_{33}\text{·CO·N(CH}_2\text{·C}_2\text{H}_4\text{)·CH}_2\text{·CH}_2\text{·}$$
$$\text{N(CH}_3\text{)(CH}_2\text{·CH}_2\text{OH)·CH}_2\text{·CH}_2\text{·O·CO·C}_{17}\text{H}_{33}\}\text{Cl}$$

are antistatic and softening agents, particularly applicable to acetate rayon. C. O. C.

Antioxidants. Standard Oil Development Co. *BP* 678,118

Bis(alkoxyhydroxyphenyl)alkanes in which the OH group is *para* to the alkoxy group are extremely effective stabilisers for many materials susceptible to atmospheric and/or thermal degradation. C. O. C.

Sizing with Nylon Solutions. British Nylon Spinners. *BP* 678,452

A size, particularly suitable for nylon, consists of a solution of a CO·NH-containing polyamide dissolved in an acid-free mixture of an aliphatic alcohol of 1–3 C and formaldehyde. C. O. C.

Protein Derivatives—Auxiliary Products. Allied Colloids (Manufacturing) Co. *BP* 678,103

Addition of a 1:2-alkyleneimine to an aqueous solution or dispersion of a protein, the pH being maintained at 4–10 and the proportion of unchanged imine never being allowed to exceed 5% on the wt. of the protein, yields compounds which are of use for treating leather, paper, and textiles. C. O. C.

Waterproof Coating Composition. Wingfoot Corpn. *BP* 677,556

A coating composition for raincoats, tents, etc. consists of a dispersion in water or an organic solvent of a water-insoluble coating material and finely divided, solid, non-porous fillers or pigments, which need not be hydrophobic. The amount of filler (in g.) per 100 c.c. of coating material is—

$$44{\cdot}87 + 21{\cdot}44\,y - 0{\cdot}938\,y^2 + 1{\cdot}625\,y^3$$

(y = wet density (lb./cu. ft.) of the filler in the dispersing medium used). The coating given by such a composition is impervious to liquid water but permits the passage of water vapour at a relatively high rate. C. O. C.

Water- and Flame-proofing Composition. Treesdale Laboratories & Textile Processing Co. *USP* 2,591,368

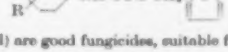
A dispersion of a chlorine-containing vinyl resin, polyvinyl chloride, or polyvinylidene chloride and antimony oxide is used. C. O. C.

Binding Agent for Printing and Padding Pastes. FBy. *BP* 676,806

A binding agent, which is soluble in water but readily convertible into an insoluble, supple, elastic film so as to yield prints which are fast to washing and do not impair the handle of the fabric, consists of polymers of esters of acrylic or subst. acrylic acid containing amino or "imino" groups, which polymers are water-insoluble but yield water-soluble salts with acids. Pastes containing such binding agents are stable for days at ordinary temperatures. C. O. C.

Furfuryl Carbanilates as Fungicides. Monsanto. *USP* 2,591,588

Compounds of formula—



(R = H or Cl) are good fungicides, suitable for application

from solution in an organic solvent or as an aqueous dispersion to textiles, leather, furs, etc. C. O. C.

Cellulose Nitrate Lacquers containing the Rosin Ester of 2 : 2 : 6 : 6-Tetramethylolcyclohexanol. General Mills. *USP* 2,592,502

2:2:6:6-Tetramethylolcyclohexanol rosin ester is entirely compatible with cellulose nitrate in presence of the usual plasticisers. Added to cellulose nitrate lacquers it results in films having more gloss and at least 50% harder without sacrifice of flexibility and abrasion resistance, nor does it increase the viscosity of the lacquer. Up to twice as much of the rosin ester may be used as of cellulose nitrate. Use of large proportions of the rosin ester has the advantages of reducing the viscosity of the lacquer and, because of the high tolerance for plasticisers, allowing production of highly flexible films. C. O. C.

Organotin Compounds—Stabilisers for Organic Materials containing Halogens. Metal & Thermit Corpn. *USP* 2,591,675

Compounds of formula $[(R^1R^2SnO)_x(OCR^3R^4)]_n$ (x = 1–6; $n > 0$; R^1, R^2, R^3, and R^4 = Alk, alkenyl, Ar, or heterocyclic radicals, and R^3 and R^4 may = H) stabilise organic materials containing halogen, e.g. polyvinyl chloride, especially to heat. C. O. C.

Whitening Agents (IV p. 469, 470).

Utilisation of Chaff and the like for the Production of Furfuraldehyde as a By-product of Flax Processing (VI p. 474).

Sizes for Paper (XI p. 481).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Extramolecular Interaction and the Colour of Organic Compounds. V. A. Izmailsky. *Eighth Aniline Dye Conference (U.S.S.R.)**, 88–120.

The quantum-mechanical basis of the theory of colour in organic compounds is outlined, and the author's views on the formation of "exo-bonds" (*J.S.D.C.*, **67**, 198–199 (May 1951)) between different molecules or between separated chromophoric systems in the same molecule are given in detail. A survey is made of the experimental basis for these views and, in particular, of work by the author and his associates on dyes containing a pair of chromophoric systems separated by an insulating chain of atoms that breaks the conjugation (~ 300 new compounds of this type were synthesised). Selected results are given in the form of tables and absorption curves. There are 59 references. A. E. S.

* See *J.S.D.C.*, **68**, 460 (Nov. 1952).

Phase Equilibria in Sulphonic Acid–Water Systems. D. Taylor and G. C. Vincent. *J.C.S.*, 3218–3224 (Aug. 1952).

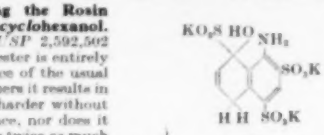
Solubility and vapour-pressure relations for the systems X·$C_6H_4SO_3H$–H_2O (X = H, *o*-CH_3, *p*-CH_3, *p*-NO_2, *p*-F, *p*-Cl, and *p*-Br) have been measured, twenty hydrates identified, and several heats of solution and of dissociation calculated. Results indicate a greater stability of monohydrates relatively to higher hydrates, in agreement with probable ionic structures for the former. H. H. H.

Some Reaction Products of 3-Chloro-4:6-dinitrobenzoic Acid. H. Goldstein and R. Stamm. *Helv. Chim. Acta*, **35**, 1470–1475 (Aug. 1952).

The chlorine atom in 3-chloro-4:6-dinitrobenzoic acid is mobile and easily replaced by other substituents when treated with nucleophilic reagents. Reduction of the above acid by stannous chloride in hydrochloric acid gives uniquely 4-chloro-1:3-phenylenediamine, whereas catalytic hydrogenation in presence of Raney nickel affords 3-chloro-4-nitro-6-aminobenzoic acid, and in no case is the diamino acid produced. H. H. H.

Bisulphite Compounds. XVI—Bisulphite Compound of 1:8-Aminonaphthol-2:4-disulphonic Acid. V. N. Ufimtsev and M. I. Chernyak. *J. Gen. Chem. U.S.S.R.*, **22**, 926–929 (June 1952).

The cpd. formed on adding a bisulphite to a soln. of 1:8-aminonaphthol-2:4-disulphonic acid is isolated as the Na or K salt. Analysis shows that one mol. of bisulphite is combined per mol. of the sulphonic acid, so that it may have either of structures I and II—



(I) (II)

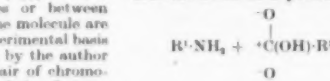
The bisulphite cpd. is stable in acid soln., but decomposes in alkaline soln. to give the original sulphonic acid. It does not couple with diazonium salts, but may be diazotised and coupled with suitable components to give azo dyes, which differ in hue from the dyes obtained by diazotising and coupling the original sulphonic acid, but are converted into these when treated with alkali. The cpd. must therefore be assigned structure I. A. E. S.

4:5-Dinitrophthalic Acid. H. Goldstein and J.-P. Merminod. *Helv. Chim. Acta*, **35**, 1476–1480 (Aug. 1952).

4:5-Dinitro-2-methylbenzoic acid and also 4:5-dinitro-1:2-xylene are readily oxidised to 4:5-dinitrophthalic acid; the last has a mobile nitro group, which is easily replaced by other substituents when treated with nucleophilic reagents such as aq. NaOH, methyl alcoholic potash, conc. ammonia, and aniline. H. H. H.

Basic Character of Amines and their Reactivity. B. A. Poray-Koshits. *Eighth Aniline Dye Conference (U.S.S.R.)**, 43–55.

The kinetics of the reactions of aromatic amines, including acylation, diazotisation, and the formation of anils, diazoamino compounds, and iminazoles (from *o*-diamines and a carboxylic acid), are studied in relation to the basic dissociation constants of the amines. All the reactions can be included under one general scheme, according to which the first stage is the formation of a bond between the unshared electron pair on the amino N and an electron-deficient atom in a polarised attacking molecule, e.g.—



The intermediate cpd. so formed splits off a simple molecule, such as H_2O or HCl, to give the final, stable product. The readiness with which such reactions occur is therefore determined by the basicity of the amine and by the readiness with which the attacking molecule is polarised. The first of these conclusions is abundantly confirmed by data given in this paper and by those of Gerchuk *et al.* and of Lastovsky (see pp. 465–466). There is no direct experimental evidence for the second, but it is considered that an examination of the catalytic action of protons on these reactions provides indirect evidence. A. E. S.

* See *J.S.D.C.*, **68**, 460 (Nov. 1952).

Effect of Certain Factors on the Rate of Diazotisation of Aromatic Amines. T. N. Vernikovskaya. *Eighth Aniline Dye Conference (U.S.S.R.)**, 56–73.

A survey is given of theoretical and practical work on the kinetics of the diazotisation reaction. 18 amines are examined experimentally. The concn. of acid (HCl or H_2SO_4) is varied from 0·05 to 4 N., the concn. of amine is 0·005 N., and half of the theoretical amount of nitrite is added. The time for complete reaction (starch–iodide indicator) is determined. In other experiments, additions of neutral salt (NaCl or KCl) are made. The reaction rate increases steadily with increase in concn. of HCl, and addition of NaCl or KCl produces a further increase in rate. It is considered that the Cl^- ion is a positive catalyst and the H^+ ion a negative catalyst for the reaction. Other factors being equal, high rate of reaction is associated with the presence of electrophilic substituents in the amine. The results over the present range of acid concentrations are contrasted with those previously obtained by the author in a lower concn. range, in which

the variation in rate amongst the amines is much less. When H_2SO_4 is used in place of HCl, reaction rates are in general lower, and with rising acid concn. the reaction rate at first falls and then rises. The results are considered on the basis of reaction between the ammonium ion and undissociated nitrous acid, and two mutually opposing tendencies are attributed to electrophilic substituents—a tendency to delay the reaction by reducing the basic character of the amine and therefore the concn. of ammonium ion at low acid concn., and a tendency to accelerate the reaction by increasing the mobility of H atoms in the intermediate product. The relative behaviour of different amines is therefore greatly dependent on acid concn.; the apparently divergent results of various workers are to be attributed largely to this circumstance. In the discussion following the paper, criticism of the experimental method as inaccurate is countered by the author. A. E. S.

* See *J.S.D.C.*, **68**, 460 (Nov. 1952).

Mechanism of the Diazotisation Reaction. B. A. Poray-Koshits. *Eighth Aniline Dye Conference* (*U.S.S.R.*)*, 74–78.

The results of Vernikovskaya (above) and of others on the rate of diazotisation are most readily interpreted on the author's general theory of the reactions of amines (p. 464), i.e. on the assumption that the primary reaction occurs between the free base and a nitroso group. Introduction of *ortho* or *para* electrophilic groups into the amine will retard the first stage of the reaction (complex formation) and accelerate the second stage (complex → diazonium ion). The effect of excess of HCl is complex. Thus, chloride ions activate nitrous acid by converting it partly into the more active nitrosyl chloride, and at high $[H^+]$ the still more active $[Cl{\cdot}N{\cdot}OH]^+$ is formed. Conversion of the intermediate complex into the diazonium ion is encouraged by chloride ions and discouraged by hydrogen ions. It is shown, in agreement with the results of various authors, that at a concn. of acid that is sufficiently low to permit the concn. of free base to be appreciable in all cases, strong bases may diazotise more rapidly than weak bases, whereas at higher acid concn., at which the hydrolysis of salts of strong bases is largely suppressed, weak bases will diazotise more rapidly than strong. Other evidence from the literature is cited in confirmation of the proposed mechanism. A. E. S.

* See *J.S.D.C.*, **68**, 460 (Nov. 1952).

Reactions with ^{15}N. V—Mechanism of the Diazoaminobenzene → *p*-Aminoazobenzene Transformation. K. Clusius and H. R. Weisser. *Helv. Chim. Acta*, **35**, 1524–1527 (Aug. 1952).

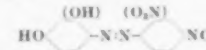
It is shown by means of ^{15}N that the transformation of diazoaminobenzene to *p*-aminoazobenzene, effected by aniline hydrochloride, takes place through a quantitative exchange between the $-N{\cdot}C_6H_5$ group of the triazene derivative and that of the aniline salt. The central nitrogen atom of the diazoaminobenzene is not concerned in the exchange. H. H. H.

Nature of the Internal Hydrogen Bond [in Azo Compounds]. A. Burawoy, A. G. Salem, and A. R. Thompson. *Chem. and Ind.*, 668–669 (5 July 1952).

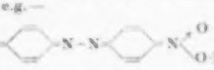
Spectroscopic observations on numerous derivatives of 1-phenylazo-2-naphthol in various solvents show that equilibria exist between the two possible tautomers, and that they vary with the solvent and the nature of substituents in the phenyl group. Details are given of selected examples—1-phenylazo-2-naphthol in hexane, alcohol, chloroform, and 50% alcohol exists as azo and phenylhydrazone tautomers, the former present in appreciable quantity only in hexane. Spectra of 1-*p*-methoxyphenylazo-2-naphthol in the four solvents show the phenylhydrazone tautomer to preponderate slightly only in dilute alcohol, the methoxyl group having stabilised the azo tautomer. Introduction of two methyl groups to give 1-(4-methoxy-3:5-dimethylphenylazo)-2-naphthol reduces the methoxyl effect; the phenylhydrazone long-wave absorption band becomes preponderant in chloroform solution. Results are held unambiguously to exclude the interpretation of substances containing an internal hydrogen bond as "resonance hybrids". J. W. B.

Structure of Hydroxyazo Dyes and their Tendency to Change Hue on a Magnesium Hydroxide Surface. L. M. Kulberg. *J. Gen. Chem. U.S.S.R.*, **22**, 969–973 (June 1952).

The author has shown that it is a general property of dyes of formula—



to react with $Mg(OH)_2$ with a change in hue, and that the product is derived from the quinonoid, nitronic acid form of the dye, e.g.—



He now shows that hydroxyazo dyes in which the NO_2 group is absent or in the *meta* position show a change in hue only when the OH is *ortho* to the azo link. These are considered to have the co-ordination structures normally attributed to this type of lake. It is found that the behaviour of an alkaline soln. of the dye on adding acetone forms a criterion of the type of Mg cpd. that the dye forms—there is no change of hue when the Mg cpd. is of the co-ordination type, and the hue changes to that of the Mg cpd. when this is a deriv. of a nitronic acid. The Mg reactions of 32 hydroxyazo dyes are tabulated. A. E. S.

Acetate Rayon Dyes. II—Poly(hydroxyethyl)aniline Dyes. H. Wahl, Y. Arnould, and M. Simon. *Bull. Soc. chim. France*, 366–369 (1952): *Chem. Abs.*, **46**, 7331 (10 Aug. 1952).

Spectra and m.p. of monoazo dyes derived from aniline and its substituted derivatives, e.g. nitro- and chloroanilines, coupled with diethylaniline, ethylhydroxyethylaniline, bishydroxyethylaniline, ethylbishydroxyethylaniline, and ethyltrishydroxyethylaniline are given. C. O. C.

Azo Dyes from 4-Amino-1-naphthylacetic Acid. Y. Ogata, M. Okano, and Y. Kitamura. *J. Org. Chem.*, **16**, 1588–1592 (1951): *Chem. Abs.*, **46**, 6117 (10 July 1952).

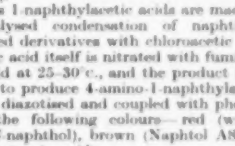
Various 1-naphthylacetic acids are made by the Fe_2O_3–KBr-catalysed condensation of naphthalene and its substituted derivatives with chloroacetic acid. 1-Naphthylacetic acid itself is nitrated with fuming nitric acid in acetic acid at 25–30°C., and the product reduced with Sn and HCl to produce 4-amino-1-naphthylacetic acid. This on being diazotised and coupled with phenols gives dyes, e.g. of the following colours—red (with α-naphthol), orange (β-naphthol), brown (Naphtol AS), violet (H acid or chromotropic acid). R. K. F.

Bromination of Compounds containing Two Aromatic Nuclei. X—Bromination of Arylamides of 5-Nitrosalicylic Acids. G. V. Jadhav and P. G. Nerlekar. *J. Indian Chem. Soc.*, **29**, 234–237 (April 1952).

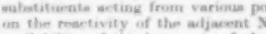
Bromination of arylamides of 5-nitrosalicylic acid when carried out in glacial acetic acid gave the corresponding 3-bromo-5-nitrosalicyloyl-bromoarylamide, except in the case of the *o*-, *m*-, and *p*-nitroanilides, when no substitution of the arylamide group took place, and in those of the *o*- and *p*-anisidides, when two bromine atoms were introduced into the arylamide part of the molecule. With liquid bromine more highly brominated compounds were formed, except in the case of the *m*- and *p*-nitroanilides and the *o*-anisidide. C. H. R.

Formation of Aryl-*N*-nitrosoureas. R. P. Lastovsky. *Eighth Aniline Dye Conference* (*U.S.S.R.*)*, 30–34.

Diarylureas $Ar^1{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}Ar^2$ are nitrosated with a soln. of nitrous anhydride in acetic acid, and the products, which are in all cases *mono*nitroso derivatives, are treated with an alkaline soln. of β-naphthol to give azo dyes—



In every case the nitrosation product yields a single azo dye, which is identified in order to indicate the point at which nitrosation occurred. The results, which are summarised below, clearly show the relative effects of various substituents acting from various positions in the nucleus on the reactivity of the adjacent NH group, i.e. on the availability of the lone pair of electrons on the amide N. The presence of nitro and carboxy groups reduces the readiness with which nitrosation occurs, and while nitrosation products are obtained from the 3:4′- and 3:3′-dinitro compounds, the 2:4:2′:4′-tetranitro cpd. does not react.



Substituent in I	Substituent(s) in II	Nitrosation occurs at
None	2-, 3-, or 4-NO_2	N^1
None	2-, 3-, or 4-COOH	N^1
None	2- or 3-Cl	N^1
None	4-Cl	N^2
None	2- or 3-CH_3	N^2
None	2:4-di-CH_3	N^2
4-NO_2	3-NO_2	N^2
4-NO_2	4-Cl	N^2

A. E. S.

* See *J.S.D.C.*, **68**, 466 (Nov. 1952).

Exchange Reactions of Urea Derivatives. M. P. Gerchuk, D. A. Livshits, and S. Z. Tayts. *Eighth Anilne Dye Conference (U.S.S.R.)**, 35–42.

Following the observation that attempts to recrystallise an *NN′*-dithiazolylurea from aniline lead to the formation of *NN′*-diphenylurea, other examples of the general exchange reaction—

$$R^1{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}R^1 + 2R^2{\cdot}NH_2 \rightleftharpoons R^2{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}R^2 + 2R^1{\cdot}NH_2$$

(R^1 and R^2 = aromatic or heterocyclic radicals) are studied. In general, the greater the basicity of $R^2{\cdot}NH_2$, the more the equilibrium is displaced to the right of the equation. A. E. S.

* See *J.S.D.C.*, **68**, 460 (Nov. 1952).

Light Absorption of some Colour Coupiers derived from *p*-Phenylenediamine. I—Relation between Light Absorption and Structure of the Azomethylene Series of Dyes from *p*-Phenylenediamine Derivatives containing the Pyrazolone Nucleus. K. Furuya and F. Ueno. *J. Soc. Sci. Phot. Japan*, **13**, (4), 27–33 (1951): *Chem. Abs.*, **46**, 7447 (25 Aug. 1952).

Absorption curves of 16 compounds are given. The first absorption maximum is near the green region, with a second, smaller maximum at shorter wavelengths. Blue transmittance increases when the benzene nucleus attached to the pyrazolone N is replaced by naphthalene or when NH_2, SO_3K, COOK, NO_2, etc. are introduced into the benzene nucleus. C. O. C.

Absorption Spectra of Di- and Tri-phenylmethane Compounds. V. V. Perekalin, M. V. Savostyanova, and R. I. Morozova. *J. Gen. Chem. U.S.S.R.*, **22**, 821–829 (May 1952).

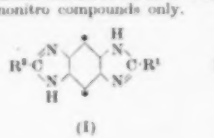
Absorption spectra (200–700 mμ) are determined in alcoholic soln. of di- and tri-phenylmethane and di- and tri-phenylcarbinol, of their *p*-dimethylamino derivatives as free bases and as salts, and of various relevant related compounds, and regularities revealed by a comparative examination of the results are discussed. A. E. S.

Fundamental Polarisation of the Luminescence of Dyes—Fluorescence and α-Phosphorescence. E. Laffitte. *Compt. rend.*, **234**, 424–426 (1952): *Chem. Abs.*, **46**, 6491 (25 July 1952).

The fundamental polarisation p_0 of the fluorescence is independent of the spectral region, values for the following dyes in solid solution in Plexiglass being—Fluorescein 0·43, Acridine Yellow 0·42, Rhodamine B 0·43, Euchrysine 0·43, trypaflavine 0·42, pinacyanol 0·47. The phosphorescence has the same spectral distribution as the fluorescence, but its degree of polarisation decreases slightly with increasing wavelength—trypaflavine 0·32, Euchrysine 0·32, Acridine Yellow 0·25. C. O. C.

Iminazole Derivatives. III—Nitration of Benziminazole Derivatives and the Preparation of some New 1:2-4:5-Bisiminazolobenzene Derivatives. IV—Chemical Properties of 1:2-4:5-Bis-iminazolobenzene Derivatives. L. S. Efros. *J. Gen. Chem. U.S.S.R.*, **22**, 1008–1015; 1015–1024 (June 1952).

In agreement with the observation of Kym and Ratner (*Ber.*, **36**, 3968 (1912)) it is shown that, in the nitration of benziminazole and its 2-methyl deriv., the second NO_2, which is introduced with some difficulty, enters the position *ortho* to the first, yielding the 5:6-dinitro deriv. The explanation now advanced for this behaviour is based on the supposition that the benzene ring of benziminazole is deformed and its conjugation disturbed, particularly at the 5–6 link, which is considered to have a bond order approaching unity. The dinitro cpd. may be reduced to a diamine, which will react with a carboxylic acid (R^2·COOH) and HCl to give a deriv. of 1:2-4:5-bisiminazolobenzene (I—R^1 = H or CH_3). A number of such compounds are prepared and their properties studied. Considerations of the type advanced above for benziminazole are applied to I and lead to the prediction of an unsaturated condition at the *meso* (*) positions, similar to that found in anthracene. I, in fact, behaves analogously to anthracene in many of its reactions. Thus, it is oxidised by chromic acid to give, in good yield, the coloured quinone II, which is only very feebly basic, but dissolves in alkalis to give a violet–blue soln. This is decolorised by hydrosulphite, but the leuco cpd. formed readily re-oxidises in the air. As in the case of anthracene, chlorination and bromination of I proceed readily in the *meso* positions, giving products that, like the original substance, are oxidised by chromic acid to the quinone. Nitration occurs also in the *meso* position, giving monnitro compounds only.



A. E. S.

Styrene and Cyanine Dyes derived from Naphtho-1:4-thiazines. F. S. Babichev. *Ukrain. Khim. Zhur.*, **16**, 188–198 (1950): *Chem. Abs.*, **46**, 5054 (10 June 1952).

2:3-Dihydro-3-methyl-2-methylenenaphtho[2:1]thiazole treated in benzene with—

CH_2·CO·CHBr·C_6H_5 or CH_3·CO·CHBr·$COOC_2H_5$

yields addition compounds from which, after separating as the perchlorates and decomposing with aqueous–alcoholic HCl, naphtho-1:4-thiazine derivatives are isolated. These on treatment with varying amounts of *p*-dimethylaminobenzaldehyde give brown, violet, and green styrene dyes on the one hand; and with ethyl orthoformate cyanine dyes on the other. Thus, the methyl perchlorate of 2-phenyl-3-methyl-7:8-benzobenzo-1:4-thiazine heated in ethanol with *p*-dimethylaminobenzaldehyde yields the green methyl perchlorate of 2-phenyl-3-*p*-dimethylaminostyryl-7:8-benzobenzo-1:4-thiazine. R. K. F.

Some Trinuclear Cyanine Dyes. V—Attempted Synthesis of Dyes isomeric with those of the Neocyanine Type. F. M. Hamer. *J.C.S.*, 3197–3211 (Aug. 1952).

Four trinuclear trimethincyanines have been synthesised by condensing a *meso*-methyltrimethincyanine with a heterocyclic aldehyde or with an anil. Other *meso*-substituted trimethincyanines have also been obtained via the known method involving 2-substituted benzoxazolium salts. A trinuclear trimethincyanine resisted quaternary salt formation. Absorption data are recorded. H. H. H.

Synthesis of Trinuclear Sensitising Dyes. T. Ogata. *J. Soc. Sci. Phot. Japan*, **13**, (2), 24–27 (1950): *Chem. Abs.*, **46**, 6531 (25 July 1952).

1-Ethyl-4-[3-(1-ethyl-1(4*H*)-quinolylidene]propenyl]-quinolinium iodide (I) was synthesised from lepidine ethiodide (II) and ethyl orthoformate (III). Mechanisms

are given showing how 4:4′-{3-[2-(1-ethyl-4(1*H*)quinolylidene)ethylidene]propenylene}bis[1-ethylquinolinium iodide] is obtained by interaction of I and III, and 1:1′-diethyl-4:4′-cyanine iodide from I and II. 2:2′-{3-[2-(3:4-Dimethyl-4-thiazolin-2-ylidene)ethylidene]propenylene}bis[3:4-dimethylthiazolium iodide] (IV) was obtained from the normal carbocyanine iodide (3:3′:4:4′-tetramethyl-2:2′-thiazolocarbocyanine iodide) (V) and III in acetic anhydride, and from V and 2-(2-anilinovinyl)-3:4-dimethylthiazolium iodide in acetic anhydride with K acetate. C. O. C.

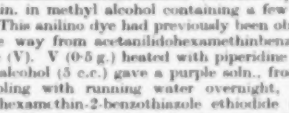
Synthesis of Polymethinaminocmicyanines. T. Ogata and K. Sugimoto. *Repts. Sci. Research Inst.* (Japan), **27**, 332–335 (1951): *Chem. Abs.*, **46**, 6837 (25 July 1952).

$C_6H_5{\cdot}N(CH_3){\cdot}[CH{:}CH]_2{\cdot}CHO$ (I) (0·1 g.) heated at 100°c. for 50 min. with 2-methylbenzothiazole ethiodide (II) (0·15 g.) in pyridine (0·5 c.c.) gave the blue methylanilinohexamethinbenzothiazole ethiodide (III).

$C_6H_5{\cdot}N(CH_3){\cdot}[CH{:}CH]_2{\cdot}CH{:}N(CH_3){\cdot}C_6H_4Cl$ (0·5 g.) was similarly condensed with II (0·3 g.) to give III (0·3 g.). I (0·19 g.) and piperidine hydrochloride (0·13 g.) in methyl alcohol (2 c.c.) for 2 hr. at 100°c., cooled, added to dil. HCl, allowed to stand overnight, the ppt. being then freed from a small amount of an insoluble substance with acetone and crystallised from dil. HCl, gave the red $C_6H_{10}N{\cdot}CH{:}[CH{\cdot}CH]_2{\cdot}N(CH_3){\cdot}C_6H_4Cl$ (IV) (0·1 g.). IV (20 mg.) and II (20 mg.) heated for 3 min. at 100°c. in pyridine with stirring gave III, which was easily converted to anilinohexamethinbenzothiazole ethiodide by heating for 20 min. in methyl alcohol containing a few drops of aniline. This anilino dye had previously been obtained in the same way from acetanilidohexamethinbenzothiazole ethiodide (V). V (0·5 g.) heated with piperidine (0·1 c.c.) in ethyl alcohol (5 c.c.) gave a purple soln., from which, after cooling with running water overnight, the blue piperidylhexamethin-2-benzothiazole ethiodide (VI) was deposited. Heating V (1·25 g.) for 10 min. at 60°c. with dipropylamine (0·35 c.c.), adding 50% K_2CO_3 (6 c.c.), and stirring for 2 min. gave 3-ethyl-2-benzothiazolepentamethin-*o*-aldehyde, which was condensed with piperidine hydrochloride to yield VI. C. O. C.

Cyanine Dyes. VII—Properties of Tetramethylthiacarbocyanines. I. I. Levkoev, A. F. Vompe, N. N. Sveshnikov, and N. S. Barvyn. *J. Gen. Chem. U.S.S.R.*, **22**, 879–886 (May 1952).

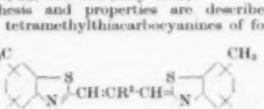
The synthesis and properties are described of 23 symmetrical tetramethylthiacarbocyanines of formula—



(R^1 = CH_3 or C_2H_5; R^2 = H, CH_3, or C_2H_5), each of the six possible arrangements of the methyl groups being represented. Introduction of an extra pair of methyl groups into a symmetrical dimethylthiacarbocyanine causes the absorption max. to undergo bathochromic displacement, which is at its greatest when the new methyl groups enter the 5:5′- or 6:6′-positions. A. E. S.

Action of Amines on certain Thiacarbocyanines containing an Alkoxy or Alkylthio Group in the *meso*-Position. N. N. Sveshnikov, I. I. Levkoev, B. S. Portnaya, and E. B. Lifshits. *Doklady Akad. Nauk S.S.S.R.*, **84**, 733–736 (1 June 1952).

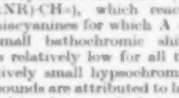
Thiacarbocyanines of formula—



(X = anion and A = O·Alk or S·Alk) react very readily with primary and secondary amines to give a new group of thiacarbocyanines (A = NH·R, NR_2, etc.), which show a hypsochromic shift, attributable to the electron-donating power of the N, of 56–89 mμ. in their absorption max. (compared with A = H). They react also with tertiary amines, Alk·X being removed with formation of symmetrical molecules containing the grouping =CH·CO·CH= or =CH·CS·CH=. For dyes in which A = NH·R, treatment with NaOH leads to loss of HX and formation of symmetrical anils (containing the grouping =CH·C(:NR)·CH=), which react with acetyl chloride to give thiacyanines for which A = NR·CO·CH_3. These show a small bathochromic shift. The max. extinction coeff. is relatively low for all the dyes. This fact and the relatively small hypsochromic shift in the non-acylated compounds are attributed to lack of planarity in the molecules arising from steric factors. A. E. S.

αγ-Polymethylenethiazolecarbocyanines—I. I. K. Ushenko. *J. Gen. Chem. U.S.S.R.*, **22**, 870–874 (May 1952).

Four new cyanine dyes of formula—



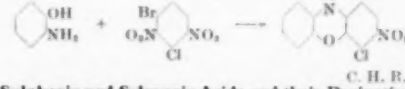
(R^1 = CH_3, R^1 = C_2H_5; R^2 = C_2H_5, R^1 = C_2H_5, *n*-C_3H_7, or *n*-C_4H_9) were prepared. They are unstable, and attempts to separate them from soln. were unsuccessful. Their absorption maxima in ethanol soln. lie at ~ 600 mμ. (~ 40 mμ. > value for the corresponding thiazolecarbocyanine without the ethylene bridge). A. E. S.

***N*-Arylsulphonylquinoneimines.** S. I. Burmistrov and E. A. Titov. *J. Gen. Chem. U.S.S.R.*, **22**, 999–1004 (June 1952).

Three of the hitherto uninvestigated *N*-arylsulphonyl-1:4-quinoneimines—$C_6H_3{\cdot}SO_2{\cdot}N{:}C_6H_4{:}O$, $CH_3{\cdot}C_6H_3{\cdot}SO_2{\cdot}N{:}C_6H_4{:}O$, and $CH_3{\cdot}C_6H_3{\cdot}SO_2{\cdot}N{:}C_{10}H_6{:}O$ (1:4-naphthaquinone deriv.)—are prepared by oxidising *N*-arylsulphonyl-1:4-aminophenols with an acid soln. of a dichromate. They are orange-yellow substances. They liberate iodine quantitatively from an acid iodide soln., giving the original aminophenol deriv., and they react readily in alkaline soln. with phenol or α-naphthol to give an indophenol (e.g. $O{:}C_6H_4{:}N{\cdot}C_6H_4{\cdot}OH$) and an arylsulphinate. A. E. S.

Trihalogenated Nitrobenzenes and their Derivatives—I. S. S. Joshi and R. S. Gupta. *J. Indian Chem. Soc.*, **29**, 193–195 (March 1952).

The 3:5-dinitro derivatives of 1:2:4-trichlorobenzene, 4-bromo-1:2-dichlorobenzene, 2-bromo-1:4-dichlorobenzene, 1-bromo-2:4-dichlorobenzene, and 1:4-dibromo-2-chlorobenzene have been prepared. With ammonia, aniline, *p*-toluidine, and piperidine, these nitro compounds gave the corresponding 2:4-diamino-1-halogeno-3:5-dinitrobenzene derivatives. With *o*-aminophenol the corresponding dihalogenonitrophenoxazine was formed, e.g.—



C. H. R.

Sulphenic and Selenenic Acids and their Derivatives. III—Formation of Selenenic Esters from Selenobromides. W. Jenny. *Helv. Chim. Acta*, **35**, 1591–1593 (Aug. 1952).

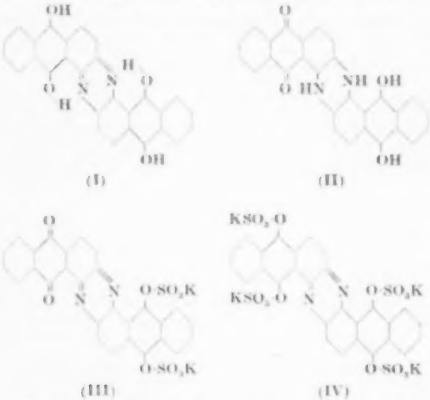
Methyl anthraquinone-1-selenenate may be obtained by the action of moist silver oxide in methanol on 1-anthraquinonyl selenobromide. This reaction establishes that the formation of selenenic esters does not necessarily require an intermediate selenyl acetate. Moist silver oxide reacts with 1-anthraquinonyl selenobromide in dioxan to form anthraquinone-1-selenenic acid. H. H. H.

Studies in Vat Dyes. IV—Preparation and Dyeing Properties of Di-imines from 2:7-Dibromophenanthraquinone. R. D. Desai and R. H. Kundel. *J. Indian Chem. Soc.*, **29**, 238–240 (April 1952).

Brown and violet vat dyes were obtained by condensing 2:7-dibromophenanthraquinone with 1- and 2-amino-, 1-amino-2-methyl-, and 1:5-diamino-anthraquinones by the Ullmann method, giving the corresponding 2:7-bisanthraquinonylaminophenanthraquinones. Similar compounds were obtained from 2:7-diaminophenanthraquinone with 1-chloro-4-acetylaminoanthraquinone and 3-bromobenzanthrone. Fastness to light of 5 to 7 and to bleaching of 2–3 was obtained. C. H. R.

Significance of the Hydrogen Bond in the Chemistry of Vat Dyes. N. S. Dokunikhin. *Eighth Aniline Dye Conference (U.S.S.R.)**, 121–134.

An account is given of well known effects, which have been discussed e.g. by Gill and Stonehill (J.S.D.C., **60**, 183–186 (July 1944)), that are attributable to hydrogen-bonding in vat dyes. A detailed discussion is given of indanthrone, of its reduction products and their sulphuric esters, and of reactions leading to the formation of these various substances. The symmetrical formula (I) for dihydroindanthrone, which on theoretical grounds (see *loc. cit.*) is preferable to the Scholl formula (II), has been found difficult to confirm experimentally, but this has now been done at Leverkusen, where the red disulphuric ester III has been prepared and found to be quite distinct from, and not directly convertible into, the violet disulphuric ester of dihydroindanthrone, which is considered to be derived from I by esterification of the outer hydroxyls. The author's work shows that the product obtained by Scholl by benzoylation of tetrahydroindanthrone is a tetrabenzoyl deriv. not of this substance but of the corresponding azine. Similarly, the tetrasulphuric ester of tetrahydroindanthrone has not been prepared directly, and the well known fluorescent yellow tetra-ester IV is derived from the azine. IV can be reduced to the ester of tetrahydroindanthrone, a brown, non-fluorescent substance that readily reoxidises in the air. It is well known that leuco vat esters require the simultaneous presence of acid and oxidising agent for development. The azine grouping is itself an oxidising agent, equivalent in its potential effect to one half of the tetra-ester molecule. Consequently, on acidifying a soln. of the usual tetra-ester, two of the ester groups are split off, giving the violet di-ester derived from I; however, if acidification is with acetic acid and is followed immediately by neutralisation with alkali, a red tri-ester can be isolated. The tri-ester may be oxidised in alkaline soln. to the red di-ester III, which on acidification in absence of oxidising agent yields indanthrone directly. Simple acidification has no action on the tetra-ester of tetrahydroindanthrone.



The effects of hydrogen-bonding in the following vat dyes and leuco vat esters are considered—Indanthren Brown BR, Anthrasol Brown BR, Anthrasol Orange IGG, Indanthren Brilliant Violets RK and BBK, and Indanthren Blue 3G. The author has shown that Anthrasol Brown BR contains only two instead of the expected six ester groups, and he assigns to it formula V.

The variations in colour and fluorescence of solutions in various solvents of dibenzanthrone and *iso*dibenzanthrone are discussed. Solvents fall into two groups according to their ability to form hydrogen bonds with the CO groups of the dyes. The variations observed are considered to be associated with the well known effect of humidity on the hue of dyeings obtained with these substances. Finally, hydrogen-bonding effects in 16:17-dihydroxydibenzanthrone and its ethers are considered, attention being given e.g. to the molecular compounds formed between the dimethyl ether (Caledon Jade Green) and phenols and to the general dependence of this diether on some such association (e.g. with water, when dyed on a textile) for its colour, which is otherwise blue. The author's postulate of a seven-membered ring formed by hydrogen-bonding in this series of dyes is criticised in the discussion that follows the paper. A. E. S.

* See J.S.D.C., **68**, 460 (Nov. 1952).

Sulphonation of Polycarbocyclic Ketones. A. M. Lukin. *Eighth Aniline Dye Conference (U.S.S.R.)**, 135–166.

The author gives a review of his work on the double compounds ("sulphonoxides") formed between carbocyclic poly*cyclo*ketones and SO_3, on their application in sulphonation, and on their possible rôle in the mechanism of sulphonation by means of oleum or sulphuric acid. Prior to this work, only the very unstable sulphonoxides of anthraquinone had been recorded. The sulphonoxides of the following compounds have now been investigated in detail—benznaphthone, benzanthrone, 4:5-9:10-dibenzopyrene-3:8-quinone, *iso*dibenzanthrone, and pyranthrone. Sulphonoxide formation is accompanied by a bathochromic effect. One SO_3 molecule is bound by each CO group, but a further molecule may be taken up without appreciable further effect on the hue. The second SO_3 is less firmly held than the first, to which it is considered to be bound; it is slowly removed when the preparation is subjected to a stream of air. Sulphonoxides are rapidly reconverted by water into the polycyclic ketone and H_2SO_4. On heating them at 170–180°C. for 2–3 hr., monosulphonic acids are obtained in good yield (~ 100% from dibenzopyrenequinone; ~ 85% from benzonaphthone, benzanthrone, and pyranthrone; ~ 80% from *iso*dibenzanthrone). Sulphates are prepared from some of the poly*cyclo*ketones, e.g. $C_{13}H_8O.H_2SO_4$ from benzonaphthone, $2C_{34}H_{14}O_2.3H_2SO_4$ from *iso*dibenzanthrone, $2C_{28}H_{14}O_2.3H_2SO_4$ and $C_{28}H_{14}O_2.H_2SO_4$ from pyranthrone. These are quite distinct in hue from solutions of the polycyclic ketones in 93% H_2SO_4. They are relatively stable to heat and do not yield sulphonic acids on heating. It is considered that, when dissolved in conc. (> 90%) H_2SO_4, the polycyclic ketone is present mainly as sulphonoxide, which constitutes an intermediate product for sulphonation in this medium and in oleum; at lower H_2SO_4 concn., the poly*cyclo*ketone is present mainly as sulphate. Spectrum data are given in support of this view. The nature of the molecular species present in sulphonation acids is discussed. 61 references are given. A. E. S.

* See J.S.D.C., **68**, 460 (Nov. 1952).

Formation and Hydrolysis of Anthraquinone-sulphonic Acids. V. V. Kozlov. *Eighth Aniline Dye Conference (U.S.S.R.)**, 167–180.

* See J.S.D.C., **68**, 460 (Nov. 1952).

Chemistry of Indanthrone. VII — Action of Piperidine on 3:3'-Dihalogenoindanthrones and the *N*-Methyl Derivatives of 3:3'-Dibromoindanthrone. W. Bradley and H. E. Nursten. *J.C.S.*, 3027–3033 (Aug. 1952).

The mechanism of the replacement of the two halogen atoms by hydrogen (cf. *J.C.S.*, 2177 (1951): J.S.D.C., **67**, 463 (1951)) is now found to involve reduction of a quinone nucleus. H. H. H.

Mechanism of the Intramolecular Condensation of *N*-Arylamides of Hydroxycarboxylic Acids. P. A. Petyunin. *J. Gen. Chem. U.S.S.R.*, **22**, 975–979 (June 1952).

Condensation in presence of sulphuric acid of *N*-arylamides of α-hydroxyearboxylic acids, e.g.—

$$C_6H_5{\cdot}NH{\cdot}CO{\cdot}CAr_2{\cdot}OH$$

to yield oxindole derivatives is shown to proceed with intermediate formation of a coloured halochromic salt,

e.g. $C_6H_4{\cdot}NH{\cdot}CO{\cdot}CAr_2{\cdot}HSO_4$, which then undergoes ring closure to give e.g. 3:3-diphenyloxindole. The *N*-arylamide of the α-chloro acid reacts similarly. 3:3-Diphenyloxindole, 3:3-bis-2':4'-dimethoxyphenyloxindole, and 3:3-diphenyl-4:5-benzoxindole are prepared in good yield by these methods. A. E. S.

Polarographic Investigation of Indolesulphonic Acids. A. P. Terentyev and L. A. Yanovskaya. *J. Gen. Chem. U.S.S.R.*, **22**, 866–870 (May 1952).

Barium salts of indole- and indolylmethane-sulphonic acids and of certain of their derivatives are examined polarographically, using a base soln. of 0·5 N-KCl with addition of HCl to pH 4·2–4·7. The half-wave potential is highest for 3-acids, lower for 2-acids, and lower still for 1-acids and for indolylmethanesulphonic acids. These observations are applied in the identification of certain sulphonation products. It is confirmed that sulphonation of 1-acetylindole gives the 2-acid and that sulphonation with oleum of 3-acetylindole occurs in the benzene nucleus. It is shown also that sulphonation of 2-methylindole by means of the cpd. formed between pyridine and SO_3 leads to the indolylmethanesulphonic acid. A. E. S.

Crystal Structure of Indigo and its Derivatives. H. von Eller. *Acta Crystall.*, **5**, (1), 142 (1952): *J. Textile Inst.*, **43**, A 600 (Aug. 1952).

This note reports the results of an examination of the crystal structure of indigo, thioindigo, and the selenium compound of indigo. C. J. W. H.

Influence of Phenylhydrazine on the Fluorescence of Chlorophyll in Solution. W. F. Watson. *Trans. Faraday Soc.*, **48**, 526–530 (June 1952).

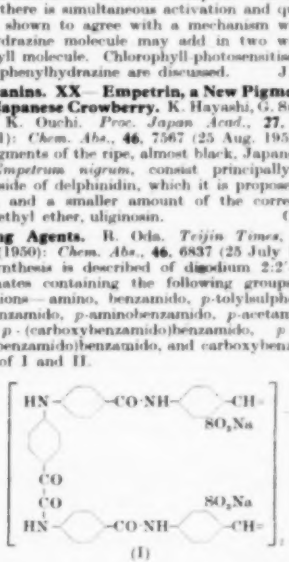
Fluorescence of chlorophyll in polar solvents is quenched by phenylhydrazine, owing to the formation, in the dark, of a non-fluorescent addition complex. In non-polar solvents there is simultaneous activation and quenching, which is shown to agree with a mechanism whereby a phenylhydrazine molecule may add in two ways to a chlorophyll molecule. Chlorophyll-photosensitised oxidations of phenylhydrazine are discussed. J. W. B.

Anthocyanins. XX—Empetrin, a New Pigment from the Japanese Crowberry. K. Hayashi, G. Suzushino, and K. Ouchi. *Proc. Japan Acad.*, **27**, 430–435 (1951): *Chem. Abs.*, **46**, 7567 (25 Aug. 1952).

The pigments of the ripe, almost black, Japanese crowberry, *Empetrum nigrum*, consist principally of the 3-galactoside of delphinidin, which it is proposed to call *empetrin*, and a smaller amount of the corresponding 3':5'-dimethyl ether, uliginosin. C. O. C.

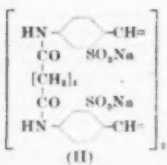
Whitening Agents. R. Oda. *Teijin Times*, **20**, (2), 1–3 (1950): *Chem. Abs.*, **46**, 6837 (25 July 1952).

The synthesis is described of disodium 2:2'-stilbenedisulphonates containing the following groups in the 4:4'-positions—amino, benzamido, *p*-tolylsulphonamido, *p*-nitrobenzamido, *p*-aminobenzamido, *p*-acetamidobenzamido, *p*-(carboxybenzamido)benzamido, *p*-(carbomethoxybenzamido)benzamido, and carboxybenzamido—and also of I and II.



(I)

and



(II)

The intensity of their fluorescence is nil to strong, affinity for cotton fair to good, and colour on cotton nil to yellow. C. O. C.

Lakes for Toning Polygraphic Colours. I. S. Vaserman. *Poligraf. Proizvodstvo*, (1), 26–27 (1948): *Chem. Zentr.*, 316 (1949): *Chem. Abs.*, **46**, 6838 (25 July 1952).

In preparing these lakes, e.g. from 2-aminonaphthalene-1-sulphonic acid→β-naphthol to form Lithol Red R or Lake Red S, it is best to add about 40% colophony soap, to replace 20% of the $BaCl_2$ usually used by $CaCl_2$, and to heat for longer than usual. A whole series of bright tones can be obtained in this way. C. O. C.

Dielectric Characteristics of Pigment Dispersions. A. Voet and L. R. Suriani. *J. Colloid Sci.*, **7**, 1–10 (1952): *Chem. Abs.*, **46**, 6451 (25 July 1951).

Observed dielectric constants of pigment dispersions are compared with theoretical values calculated from Boettcher's equation for dielectric constant as a function of volume concentration. The predicted linear relation was observed at concn. < 5% for Milori Blue (*C.I.* 1288) and Chrome Yellow (*C.I.* 1270), at < 10% for Monastral Blue B (Cu phthalocyanine) and resinated barium lithol (Ba lake of *C.I.* 180), and at < 20% for Benzidine Yellow (a benzidine→an acetoacetarylide) and unresinated barium lithol. The observed slopes of the linear plots were in only fair agreement with the calculated values, probably because the particles were not spherical as assumed in Boettcher's theory. C. O. C.

Composition of Complex Ferrous Ferrocyanide by Conductometric Method. A. K. Bhattacharya and R. S. Saxena. *J. Indian Chem. Soc.*, **29**, 284–292 (April 1952).

Conductometric titrations between ferrous sulphate and potassium ferrocyanide at various concentrations give the composition of complex ferrous ferrocyanide as being $K_2Fe^{2+}[Fe^{2+}(CN)_6]$, which with an excess of ferrous sulphate becomes $Fe^{2+}{}_2[Fe^{2+}(CN)_6]$, whereas the reverse titrations suggest that only $K_2Fe^{2+}[Fe^{2+}(CN)_6]$ is formed. The discrepancy between the calculated and the observed results can be explained by adsorption of Fe^{2+} and $[Fe(CN)_6]^{4-}$ ions. The addition of alcohol during the titrations decreases the adsorption of these ions and thus gives better results. C. H. R.

Composition of Berlin Green (Complex Ferric Ferricyanide) by Thermometric Method. A. K. Bhattacharya and R. S. Saxena. *J. Indian Chem. Soc.*, **29**, 263–269 (April 1952).

The composition of Berlin Green is ferric ferricyanide $Fe^{3+}[Fe^{3+}(CN)_6]$, as shown by thermometric titrations between ferric chloride and potassium ferricyanide at several concentrations when carried out in aqueous and aqueous–alcoholic media. The slight discrepancy between the theoretical and experimental results is explained by hydrolysis and adsorption of ferric and ferricyanide ions by the complex. C. H. R.

Cadmium Lithopones. E. G. Moss and U. Cuppini. *Pitture e Vernici*, **6**, 141–142 (1950): *Chem. Abs.*, **46**, 7340 (10 Aug. 1952).

In preparing Cd lithopone by the reaction $BaS + CdSO_4 = CdS + BaSO_4$, the pigment is obtained by mixing 0·25 M-BaS and M-$CdSO_4$ at 25°C. Na_2S can be added if the product has to contain excess CdS. The product should be dried at 250–270°C, in a current of N_2. CO_2 is unsuitable as it decomposes the CdS. Some reddening occurs during drying. Works-scale plant is illustrated. C. O. C.

History of the Manufacture of Bronze Pigments. H. Rabaté. *Trav. Peinture*, **5**, 365–368 (1950): *Chem. Abs.*, **46**, 7339 (10 Aug. 1952).

Metallic powders of various colours were used in making medieval illuminated manuscripts. Further development was carried out in Germany. Much work was done in the 19th century by H. Bessemer, and it was he who realised the necessity for producing the pigments in flake form. A table is given of the constituents of various bronzes, some of which contain (in addition to Cu and Zn) Fe, Sn, and Al. C. O. C.

Structure of Lamp Black. F. Endter. *Z. anorg. allgem. Chem.*, **263**, 191–199 (1950): *Chem. Abs.*, **46**, 7392 (25 Aug. 1952).

Hexagonal particles of lamp black observed with an

electron microscope are not compatible with the conception that the borders of the particles should be random. The anomaly can be explained by assuming the structure to consist of primary and secondary particles. C. O. C.

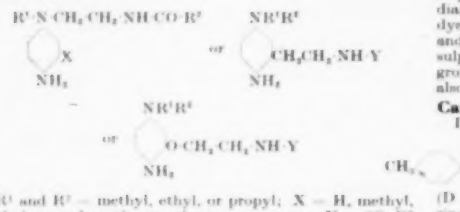
New Inorganic Pigments. E. Korinth. *Angew. Chem.*, **64**, 265–269 (1952); *Chem. Abs.*, **46**, 7341 (10 Aug. 1952).

The following new systems are described— Rinman's green and analogous compounds; the ternary systems ZnO–CoO–SiO_2, NiO–ZnO–TiO_2, CoO–Al_2O_3–TiO_2, ZnO–FeO–TiO_2, CoO–MgO–Be_2O_2 [*sic.*], etc.; manganese blue; and Pb cyanamide as an anti-rust pigment. C. O. C.

PATENTS

p-Phenylenediamine Derivatives having greatly Reduced Allergenic Properties. Eastman Kodak Co. *USP* 2,592,363–4

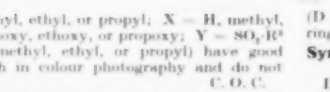
Compounds of formula—



(R^1 and R^2 = methyl, ethyl, or propyl; X = H, methyl, ethyl, propyl, methoxy, ethoxy, or propoxy; Y = $SO_2{\cdot}R^3$ or $CO{\cdot}R^3$; R^3 = methyl, ethyl, or propyl) have good developing strength in colour photography and do not irritate the skin. C. O. C.

Non-diffusing Acyl-J Acid Colour Formers. General Aniline. *USP* 2,591,642

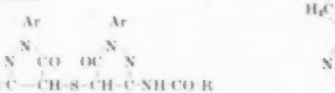
Compounds of formula—



(A = an aromatic radical of the benzene, naphthalene, pyridine, or quinoline series; X = CO, $O{\cdot}CH_2{\cdot}CO$, $CH_2{\cdot}CO$, or $NH{\cdot}CH_2{\cdot}CO$; Y = Alk of > 9 C or an aromatic radical containing an aliphatic chain of > 9 C; M = H or a salt-forming radical, e.g. Na or K; R = Alk of < 4 C) are colour formers for the cyan image in the azine method (*USP* 2,486,440: *J.S.D.C.*, **66**, 298 (1950)) and are more stable than the corresponding long-chain urethans of acyl-J acids. C. O. C.

Thiobis(acylaminopyrazolone) Colour Couplers. Eastman Kodak Co. *USP* 2,592,303

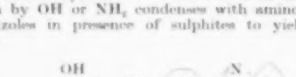
Compounds of formula—



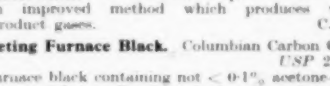
(R·CO = acyl) are colour couplers for producing magentas. C. O. C.

Azo Dyes derived from 1-Naphthol-3-sulphonic Acid carrying a Benzothiazolylphenylamino Residue as Substituent. Ciba. *BP* 677,204

1-Naphthol-3-sulphonic acid substituted in the 6- or 7-position by OH or NH_2 condenses with aminophenylbenzothiazoles in presence of sulphites to yield compounds—



(the phenylene rings A and B may contain substituents, notably CH_3 or SO_3H) which couple with diazo compounds to give azo dyes. Copperable direct cotton dyes are made by employing suitable diazo compounds. Thus 1:6-dihydroxynaphthalene-3-sulphonic acid is condensed with dehydrothio-*p*-toluidinesulphonic acid in presence of sodium bisulphite to yield—



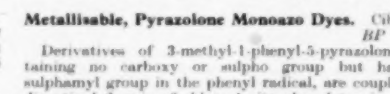
which is coupled with the diazo compound of the aminoazo compound 5-aminosalicylic acid → 2:5-dimethoxyaniline. The disazo dye so formed gives greenish blues on cotton by a single-bath or two-bath aftercoppering process. E. S.

Metallisable, Pyrazolone Monoazo Dyes. Ciba. *BP* 678,492

Derivatives of 3-methyl-1-phenyl-5-pyrazolone, containing no carboxy or sulpho group but having a sulphamyl group in the phenyl radical, are coupled with diazotised 2-amino-6-chloro-4-nitrophenol to give orange dyes which may be metallised in substance or on the fibre, and are particularly suitable for metachrome dyeing. The sulphamyl group may carry *N*-alkyl or *N*-hydroxyalkyl groups, and substituents such as CH_3, O-Alk, or Hal may also be present in the phenyl radical. E. S.

Carbocyanine Dyes. Ilford. *BP* 678,626

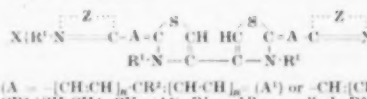
Dyes of formula—



(D = atoms to complete a 5- or 6-membered heterocyclic ring; n = 0 or 1) are photographic sensitisers. C. O. C.

Symmetrical Tetranuclear Cyanine Dyes. General Aniline. *USP* 2,592,515

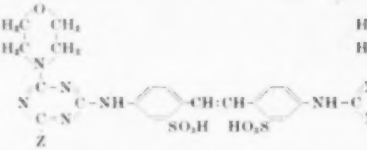
Dyes of formula—



(A = $-[CH{\cdot}CH]_n{\cdot}CR^2{\cdot}[CH{\cdot}CH]_m-$ (A^1) or $-CH{\cdot}[CH{\cdot}CH]_n{\cdot}CR^3{\cdot}[CH{\cdot}CH]_p{\cdot}CH-$ (A^2); R^1 = Alk or aralkyl; R^2 = H or Alk, but only = H when n = 1 and A = A^1; n = 1 or 2; X = an anion; Z = atoms to complete a 5- or 6-membered ring) are produced by treating 1 mol. of a diquaternary *cyclo*ammonium salt of 2:2′-dimethyl-4:4′-bisthiazole with 2 mol. of a quaternary *cyclo*ammonium cyanine dye intermediate. C. O. C.

Whitening Agents from *pp*′-Bis-[1:3:5-triazinyl-aminostilbene-oo′-2:2′-disulphonic Acid. General Aniline. *BP* 678,291

Compounds of formula—



(Z = chloro- or sulpho-anilino or morpholino) are excellent optical bleaching agents of good fastness to chlorine and light. C. O. C.

Furnace Black. J. M. Huber Corpn. *BP* 678,243

An improved method which produces valuable by-product gases. C. O. C.

Pelleting Furnace Black. Columbian Carbon Co. *USP* 2,591,024

Furnace black containing not < 0·1% acetone-extractable material is pelleted, and then heated in a still, unrenewed atmosphere to a temperature above the softening point of the acetone-extractable material but below that at which coking occurs, heating being discontinued while a substantial proportion of the acetone-extractable material remains and the pellets being cooled in a still atmosphere to below oxidation temp. This gives strong pellets, which, however, are readily milled and in which the dispersibility of the black remains unaffected. C. O. C.

Aluminium Paste Pigment. Aluminum Co. of America. *USP* 2,591,245

A product of improved stability, brightness, and specular reflectivity is obtained by subjecting a mixture of 68–85% Al flake coated with a leafing agent in presence of 14–28% mineral spirits containing dissolved stearic acid to a wet polishing operation. This is done by adding 1–4% of solid stearic acid (on the wt. of Al) and stirring so that flake rubs against flake for 1–12 hr. at < 110° F. C. O. C.

Iron Blue Pigments. DuP. *USP* 2,592,169

Improved texture and tinctorial properties are obtained if the white intermediate is acidified and digested in a closed vessel while subjected to vigorous mechanical agitation (requiring > 1 h.p./1000 gal. (U.S.A.) of suspension) and to oxidising conditions which convert not > 6% of the precipitate to the blue, oxidised state. The whole is then oxidised to the final blue pigment while in aqueous suspension. C. O. C.

Titanium Dioxide. DuP. *USP* 2,591,988

The whiteness of pigment-developed anhydrous TiO_2 is improved by dry-grinding it with 0·1–2·0% by wt. of white silica or alumina aerogel in a fluid-energy type of mill until the desired smooth-textured pigment fineness is obtained. C. O. C.

Stability of Orange Pigments (V below).

Influence of Acidity on the Absorption Spectrum of some Vat Dyes and its Relationship to the Redox Potential (VIII p. 478).

Evaluation of Aluminium Pigments (XIV p. 482).

V—PAINTS; ENAMELS; INKS

Stability of Orange Pigments. H. B. Gough *et al. Official Digest Federation Paint & Varnish Production Clubs*, (323), 863–866 (1951): *Chem. Abs.*, **46**, 7786 (25 Aug. 1952).

Outdoor exposure durability studies were made with C.P. Medium Chrome Orange, C.P. Molybdate Orange, Benzidine Orange Toner, Dinitraniline Orange Toner, Cadmium Lithopone Lt. Red, and Sb_2S_3. They were made up into 100% colour enamels, 50% colour–50% white pigment enamels, and light tints of approx. the depth of colour of 10 Medium Chrome Orange and 100 white pigment. In all cases the vehicle was an alkyd of Z1–Z2 viscosity, containing 36% phthalic anhydride, 52% linseed oil, and 50% solids in mineral spirits. All enamels had a 15% pigment vol. concn. and a 42% non-volatile content. They were exposed at 45° facing south in Florida and Delaware, and were examined every three months for one year. Colour retention of the light tints of all the pigments except the Medium Chrome Orange was very poor. The 100% colour enamel made from Hansa Orange Toner showed no colour change after 1 yr. With 50% orange pigment content Hansa Orange Toner (considered the best pigment of those tested) showed slight fading and mediocre gloss retention. C. O. C.

Practical Aspects of Pigment Dispersion. *Official Digest Federation Paint & Varnish Production Clubs*, (316), 255–297 (1951): *Chem. Abs.*, **46**, 7341 (10 Aug. 1952).

Physical dispersion is discussed. In the shearing type of pigment dispersion, a high order of strength of vehicle is desirable. For pebble-mill dispersion, mobility of vehicle is desired. This can be obtained by reducing the strength of vehicle or by diluting with solvents. TiO_2 was the pigment used, and chemical modification, e.g. with Al_2O_3, improved dispersion. C. O. C.

Influence of the Adhesion of Printing Inks on their Properties. N. G. Goloshkevich. *Poligraf. Proizvodstvo*, (3), 315–316 (1948): *Chem. Zentr.*, 315–316 [*sic*] (1949): *Chem. Abs.*, **46**, 6847 (25 July 1952).

The behaviour of inks in printing is determined entirely by their adhesive properties, which are determined in turn by the degree of polymerisation of the varnish and the moistness of the pigments. A very simple laboratory apparatus for determining adhesive strength is described. C. O. C.

Ink-drying Problems. A. C. Healey. *Amer. Ink Maker*, **27**, (12), 26–28, 51 (1949): *Chem. Abs.*, **46**, 7341 (10 Aug. 1952).

Poor drying can be improved by adding driers or a synthetic-resins varnish. Chalking may be due, among other causes, to pigments not being easily wetted or to the medium being too thin. It is most likely to occur on very absorbent papers. Uneven drying, excessive driers, and drying on the press are discussed. C. O. C.

PATENTS

Stabilised Ready-mixed Aluminium Coating Composition. Sherwin-Williams Co. *USP* 2,591,646

Addition of < 10% of plaster of Paris to aluminium paints prevents gassing and consequent losses in storage and leafing qualities. C. O. C.

Moisture-setting Printing Inks. Coates Brothers & Co. *BP* 678,408

Addition to inks containing unmodified alkyd resins of dihydric alcohols, which contain 6–18 C (> 5 C in a straight chain) and in which the OH groups are either primary or secondary and are separated by > 4 C, confers improved press-stability and results in inks which yield films of improved "finish" and resistance to alkali, rubbing, and scuffing. C. O. C.

Cellulose Nitrate Lacquers containing the Rosin Ester of 2:2:6:6-Tetramethylolcyclohexanol (III p. 464).

VI—FIBRES; YARNS; FABRICS

Capillary Phenomena in Assemblies of Fibres. J. M. Preston and M. V. Nimkar. *J. Textile Inst.*, **43**, T 402–T 422 (Aug. 1952).

Retention of water in fibrous assemblies under different hydrostatic tensions is measured by connecting the moist fibres by an intermediate system of fine capillaries (e.g. a sintered-glass porous plate) to a manometer and suction device. Several different types of fibres are studied, and it is shown that the amount of moisture retained at a tension of 30 cm. of Hg is similar to that obtained after centrifuging at 1000 *g* for 5 min. Wool is an exception, the suction retention being three times that of hydro-extraction; use of a wetting agent of surface tension 36 dynes/cm. brings the values almost equal. Comparative measurements are made of the water content–time relation at the top of a long enclosed viscose rayon strip, starting wet and with the bottom dipping in water. The effect of wet fibre–fibre contact, with a fine intermediate capillary space, is studied by measuring fibre-fibre adhesion by means of a torsion balance at different humidities. Results are discussed, together with a theoretical approach; abnormal results for wool are considered to be due to the natural crimp, scaly surface, and hydrophobic character. J. W. B.

Movement of Water Vapour through Textile Materials. N. Gralén and G. Werner. *Bull. Inst. textile France*, (30), 379–386 (Jan. 1952).

The water vapour passes mainly through the pores of the fabric, but passage across the fibres also occurs to some extent. As the porosity of a fabric increases, passage across the fibres decreases. In the case of nylon and Orlon materials the water vapour can travel along the thin water layer adsorbed on the surface of the fibres. It was observed that nylon cloth had a much higher coefficient of permeability than could be expected from its porosity. In certain cases, but not always, the permeability returns to normal after laundering or extraction with alcohol and ether. G. L.

Peirce's Two-phase Theory of Water Absorption by Cellulose. J. W. S. Hearle. *J. Textile Inst.*, **43**, T 354 (July 1952).

A modification is suggested to Peirce's theory of the absorption of water by cellulose (*ibid.*, **20**, T 133 (1929)), on the basis that cellulose is divided into crystalline and amorphous regions; agreement with experiment remains provided that all three OH groups per hexose unit in the amorphous phase serve as adsorption sites. J. W. B.

Morphology of the Cotton Fibre. IV—Expression of Experimental Results in the Form of a New, Improved Fibre Model. W. Kling and H. Mahl. *Melliand Textilber.*, **33**, 829–832 (Sept. 1952).

A new model of the cotton fibre, derived from the results of the authors' electron-microscopical investigations (see J.S.D.C., **67**, 285 (July 1951); **68**, 224 (June), 277 (July 1952)), is described, and comparative diagrams are given

of this and other models that have been proposed. There is no evidence of a cuticle, distinct from the primary wall. The latter, which is estimated to be < 0·2 μ. in thickness, contains only ~ 10% of cellulose and consists mainly of an apparently structureless mass of wax and pectin. In this mass, which presents an unbroken, smooth surface, the cellulose is embedded in the form of two concentric layers, each consisting of a network of fibrils. The orientation of the fibrils is determined not by the direction of the fibre axis, but by the local orientation of the fibrils in the underlying secondary wall, which follow the well known periodically reversing spiral arrangement. The outer layer of cellulose is predominantly longitudinal in orientation, individual fibres being oriented at 20–30° on either side of the spiral line. It is considered that this layer is in existence during the rapid longitudinal growth of the cell. The inner layer of the primary wall is predominantly transverse in orientation, the fibrils being oriented at about 80° on either side of the spiral line, and it is considered to be formed when the rapid phase of longitudinal growth is complete. The secondary wall contains ~ 95% of cellulose and is built up in the fashion already described (*loc. cit.*) from groups of fibril bundles in the form of strands or ribbons, with intervening spaces that have a more open structure. Electron-microscopical examination shows no signs of a concentric arrangement of fibril bundles or of other evidence of daily growth rings. There is no doubt about the evidence for the deposition of cellulose in layers during growth and about the effect of the daily cycle, but it is considered that during the drying of the hair the structure is compacted, and all traces of the original structure are lost. Chemical attack on the fibre tends to proceed radially through less resistant places in the structure, and there are no signs of a breakdown into layers. The layered appearance observed in highly swollen fibres after pressing under a microscope cover slip, previously regarded as daily growth rings, may owe its origin to other causes, e.g. to a periodic, Liesegang type of effect in the swollen gel. For similar reasons, suggestions by earlier workers of the existence of adjacent concentric layers in which the spirals run in opposite senses are discounted. A. E. S.

Distribution of Ions between Cellulose and Electrolyte Solutions. J. Farrar and S. M. Neale. *J. Colloid Sci.*, **7**, 186–195 (1952); *Chem. Abs.*, **46**, 7762 (25 Aug. 1952).

Determination of the cationic and anionic distribution ratios between (i) Cellophane, cotton, oxycellulose, and dyed and esterified Cellophane, and (ii) aq. $Na_2S_2O_3$, NH_4Cl, and the alkali-metal chlorides showed that the ionic distribution was in general agreement with the Donnan theory if water is regarded as the solvent in both phases. In conc. soln. specific affinity between salt and cellulose must be considered. A linear relation between water retention of fibres and the surface tension of aq. soln. was observed for Cellophane, viscose and acetate rayons, wool, Vinyon, and alginate and casein fibres.
C. O. C.

Effect of High Temperature on the Physico-mechanical Properties of Cellulosic Fibres. T. Finkelstein, W. Kargin, and S. Rogowin. *Textil-Praxis*, **7**, 641–644 (Aug. 1952).

The effect of heating viscose-rayon and cotton fibres in a high vacuum for 2 hr. on their physical, chemical, and mechanical properties becomes appreciable only at temp. > 200° C. After prolonged heating at 240° C. under these conditions, viscose rayon loses 1 mol. of water per glucose residue and becomes insol. in cuprammonium soln. On heating acetate and viscose rayons *in vacuo* at > 200° C., the degree of polymerisation falls at about the same rate in each case. A. E. S.

Meteorological Effects on the Stress–Strain Relation of Rayons and Perlon. E. Luyken and W. Wegener. *Melliand Textilber.*, **31**, 822–825 (Dec. 1950); **33**, 767–770 (Aug.), 876–878 (Sept. 1952).

Simultaneous observation of weather conditions and of the load required to stretch a yarn (viscose, cuprammonium, or acetate rayon, Perlon, or Sedura) to a given extent in a conditioned room shows that there are factors, other than ambient temp. and R.H., that significantly affect the results, which show variations, of 4% or more from the mean, attributable to variations in the time and date of the experiment. These variations can be correlated with the variation in the temp. and pressure of the air out-of-doors, and, particularly, with conditions existing in the atmosphere at a height of 5–8 km. Daily and monthly rhythms are to be observed in the results, particularly in those for viscose and cuprammonium rayons. A. E. S.

Influence of the Chemical Constitution of Cellulosic Raw Materials on the Properties of Rayons manufactured therefrom. C. Centola. *Bull. Inst. textile France*, (30), 131–139 (Jan. 1952).

The cellulosic macromolecules contain centres which are easily ruptured by chemical means, but it must not be assumed that such weak points are necessarily eliminated in the course of the various manufacturing processes. It is probable that several properties of rayons, e.g. ageing, result from the chemical structure of the raw material and the methods employed for its purification. G. L.

Physicochemical Study of Structural Modifications of Viscose Rayon during Stretch-spinning. J. Pinte, P. Rochas, and J. Henno. *Bull. Inst. textile France*, (30), 141–176 (Jan. 1952).

The following conclusions were drawn when the extension had the values quoted—(*a*) at 0–10%, the extent of intermolecular contact in the amorphous regions is enhanced and hence the degree of order increased; (*b*) at 10–30%, a rapid increase in orientation around the fibre axis occurs; (*c*) from 30% onwards a slow but steady increase in the extent of both order and crystallinity takes place. The ratio of orientation in the skin of the fibres relative to that in the core is reasonably constant, and it is independent of the amount of stretch imparted during spinning. G. L.

High Friction of Extracted Continuous-filament Yarn on Clean Glass Surfaces. E. Bradbury and A. Reicher. *J. Textile Inst.*, **43**, T 350–T 353 (July 1952).

Frictional properties of clean untwisted Terylene, nylon, viscose rayon, acetate rayon, and silk yarns are measured by passing the yarns over a clean glass cylinder, and strikingly high frictional values are found. Thus when a yarn carrying a 50-g. weight is held at one end and passed over the cylinder, the yarn does not slide off on releasing the hold. If, however, the cylinder is exposed to the air for 1 hr. the yarn cannot be made to stay on. The effect is independent of the type of yarn, of a charged atmosphere, and over a wide range of humidities, but is prevented by using a ground-glass surface, or a twisted or staple yarn. It is suggested that the true area of contact is not proportional to the normal pressure and is influenced by factors such as the geometrical configuration of the surfaces in contact and the irreversible plastic deformation of the contact area. J. W. B.

Swelling and Swelling Anisotropy of Rayon. J. Pinte. *Melliand Textilber.*, **33**, 699–704 (Aug. 1952).

An investigation is made into the effect of spinning conditions and of subsequent processes on the swelling and swelling anisotropy properties of cellulosic rayons. The water imbibition during swelling is determined indirectly by treating the swollen rayon with diazomethane and measuring the degree of methylation. Samples of each rayon are conditioned in moist atmospheres (R.H. varied); these contain imbibed water only. Other samples are centrifuged from the wet condition; these contain both imbibed and capillary water. The samples are treated with diazomethane, and for each rayon the degree of methylation is plotted against the total uptake of water. For the centrifuged samples, the degree of methylation follows a horizontal line (const. imbibition); while for the conditioned samples it rises at first very slowly (0–15% regain) from a low value at zero regain, then rapidly (15–25% regain), and finally more gradually and linearly. The causes of this behaviour are discussed. The horizontal line at high water uptake and the final linear portion of the curve for low uptake are extrapolated, and the uptake at the point of intersection (which varies from 67 to 73% among the rayons examined) is taken as the imbibed water uptake for the fully swollen fibre. For rayons that differ only in the degree of stretch during spinning, or for samples of a given rayon that have been subjected to different chemical treatments, the capillary

water remaining after centrifuging is found to be substantially const., and it is established that the results of the centrifuge method can be used directly for the present, comparative purposes. The longitudinal swelling of the fibre, which is measured by direct microscopical observation, varies linearly with moisture regain until the latter approaches a critical value of 40–45%, above which no further longitudinal swelling occurs. The author has previously attributed this effect to the restrictive action of the "skin" of viscose rayon, but now finds that the behaviour of cuprammonium rayon, which has no skin, is similar. It is considered, therefore, that the effect is attributable to restrictions imposed by linkages between the macromolecules. The transverse swelling, which is determined indirectly from density measurements, varies linearly with regain. The ratio of the slopes of the curves for transverse and longitudinal swelling (against moisture regain) is termed the differential swelling-anisotropy coeff. C. It is shown that C is closely related to the extension on wetting A, which is defined as the % increase in length on wetting of a fibre that has been conditioned at 65% R.H. The relation found ($C = 15{\cdot}4/A$) enables a close estimate of the swelling anisotropy to be made from very simple measurements. It is found that, with increase of the stretch of the rayon during spinning, imbibition diminishes and anisotropy increases. After treating the rayon with steam (95°C.) or with hypochlorite soln., there is reduced imbibition and slightly reduced anisotropy. Treatment with steam at 110°C. leads to a still greater reduction in imbibition, but the anisotropy coeff. is not affected. The results are interpreted in terms of orientation and cross-linking effects. A. E. S.

Relationship between Structure and Creasing Properties of Viscose Rayon—II. E. Elod and M. Kramer. *Textil-Praxis*, **7**, 623–625 (Aug. 1952).

In further work on the crease-resistance of unresinated viscose rayon (cf. *J.S.D.C.*, **67**, 163 (April 1951)), it is shown that, when conditioned rayon is heated or is wetted and then dried at various temp., the recovery angle falls, and there is a rise during reconditioning, which is far from complete in 7 days. Wet rayon and the same rayon after drying at 40 or 100°C. are conditioned together. At first, the recovery angle is much greater for rayon conditioned from wet; but, in each case, the recovery angle and the elastic modulus of the fibre continue to rise long after moisture equilibrium has been substantially attained; and finally, after about 30 days, they become practically the same for all the rayon samples, in spite of the higher equilibrium moisture content of the sample conditioned from wet. Rayon treated with Kaurit DD, a modified urea–formaldehyde condensate, has a much higher initial recovery than unresinated viscose heat-treated in the same manner, but the difference in recovery is much less after a 60-day conditioning. Microscopical staining tests on resinated rayon show that the "skin" is practically free from resin. By varying spinning conditions, rayons are prepared without skin, or with skins of various thickness, and it is shown that, as the skin becomes thicker, the crease-resistance of the unresinated rayon rises, and the amount of resin fixed in the fibre falls, while the improvement in crease resistance effected by resinating declines rapidly. A. E. S.

Effects arising from the use of Titanium Dioxide Delustrant in Rayons. G. Schwen. *Melliand Textilber.*, **33**, 522–525 (June); A. Agster and F. Walz. *Ibid.*, 739–741 (Aug.); Discussion. *Ibid.*, 741–743 (Aug. 1952).

A report is given of a discussion held at the 1952 Congress of the V.T.C.C. on effects attributable to the delustring of rayons with TiO_2. Two papers were read, and these were followed by six shorter contributions. In view of the large number of complaints that arise in commercial practice from the catalytic action of TiO_2 on the photochemical degradation of cellulose and cellulose acetate rayons and of dyes, Agster and others strongly recommend that the use of matt rayon in curtains should be discontinued until the methods of combating the deleterious action have been perfected or have come into general use. Agster points out that the initial wet strength of matt rayon is generally 10–15% less than that of comparable bright rayon, and that this difference may be increased somewhat during finishing operations and further increased to 25–30% during the exposure to light that occurs before the first laundering, in which the damage generally makes itself apparent. The susceptibility of the fabric to damage by photochemical action can be reduced by using higher filament and yarn denier and by incorporating carrier fibres, particularly Orlon (but not Perlon or silk, which are themselves very susceptible to photodegradation). Also, the use of matt rayon can often be avoided by the use of one of the less lustrous cuprammonium or high-tenacity rayons. The rate of destruction of the fibre or of dyes present in the fibre can be reduced by treating the TiO_2 pigment or the matt rayon with anticatalysts (Cr, Mn, or Sb compounds; e.g. 0·1% Cr_2O_3 on the wt. of TiO_2); it is reduced also by increasing the grain size of the TiO_2. The effect of the physical properties of the pigment is discussed by F. Werther, who points out that rutile is a much less active catalyst than the usual anatase and has greater covering power; it is, however, much harder, and the delustred rayon has an abrasive action on thread guides and needles. The possibility of its general use for delustring is at present the subject of large-scale trials. Schwen's results, contrary to the findings of other workers, indicate that the loss in dry strength of viscose rayon on exposure to daylight is not appreciably affected by the presence of TiO_2, though measurements of the degree of polymerisation reveal that there is indeed some increase in degradation. Schwen recognises the need to do further work at controlled humidity. The great effect of moisture on the catalytic action of TiO_2 is stressed by a number of the participants; thus, E. Köster states that, in absence of moisture, there is no difference in the rates of fading of dyeings on bright and on matt rayon, whereas in presence of moisture, dyes vary greatly in their susceptibility to the catalytic action of TiO_2. Some dyes, e.g. Congo Orange G, may be destroyed completely during the process of drying the dyed fabric in the sun. Photochemical effects on TiO_2-delustred acetate rayon may be very great, and specific complaints of degradation and dye fading are discussed by J. Müller. H. Reumuth describes a microscopical examination of photochemically degraded matt viscose and acetate rayons. The pigment particles are found to be surrounded by extensive oxidation zones, which are made visible by various colour reactions. When the zones are large, or the particles close together, the fine structure of the rayon may be so greatly disturbed that its strength falls to zero. Degradation of rayons during washing was discussed also. Schwen and Agster obtain negative results with commercial washing agents, including those which contain oxidants; but Agster obtains increased degradation on matt viscose rayon with a commercial agent that contains ~ 50% of a perborate. J. Müller obtains similar results in oxidative wet treatments on acetate rayons, and he shows that the difference in the degradation of bright and matt rayon can be eliminated by the use of anticatalysts (Mn and Cr). A. E. S.

Special Case of Photodegradation of Delustred Viscose Rayon. J. Pinte and P. Rochas. *Bull. Inst. textile France*, (31), 9–17 (Feb. 1952).

The results of a systematic investigation of a fault are given. Stains and bars were found after dyeing with direct dyes. The fault did not occur on bright rayon, but was particularly pronounced after contact with wash liquor over a week-end. It was concluded that exposure to light of TiO_2-delustred viscose rayon immersed in tap water from various sources resulted in the formation of appreciable quantities of oxycellulose. Chemically pure water causes far less deterioration. The photochemical oxidation can be prevented either by destruction of the traces of chlorides present in tap water or by addition of a reducing agent, e.g. quinol. G. L.

Fungal Decomposition of Jute Fibre and Cellulose. IV—Action of some Physical and Chemical Agencies on Subsequent Fungal Decomposition of Jute. S. N. Basu and S. N. Ghose. *J. Textile Inst.*, **43**, T 355–T 361 (Aug. 1952).

Jute is found to be a very unstable fibre; mild action by various chemical and physical agents, such as those encountered in processing and use, greatly increases the susceptibility to fungal attack. An exception is pretreatment with fungi, which renders jute more resistant to further fungal treatment, presumably because any fungus-attractive by-products are used up as fast as they are

formed. Heat has little effect, though results suggest that prolonged storage under warm conditions may lead to increased mildew action. In contrast to cotton and jute α-cellulose, which are rendered fungus-resistant by light and oxidation to oxycellulose, jute becomes much more susceptible to fungi after attack by light, and also by alkali. This is believed to be partly due to removal of lignin, which confers resistance, but results also show that sensitising by alkali cannot be explained solely by this loss.
J. W. B.

Transverse Swelling of Jute Fibre in Water. M. M. Roy and M. K. Sen. *J. Textile Inst.*, **43**, T 396–T 401 (Aug. 1952).

Measurements are made of the transverse swelling in water of single fibres of raw, delignified, and alkali-treated jute. Delignification to 8·89% lignin content produces no appreciable change, but beyond this a considerable increase in swelling—37·7% for 0·78% lignin—shows that lignin acts as a swelling depressant. This is considered to support the view that lignin–cellulose bonds are strong. Alkali treatment also increases swelling, presumably by rupture of alkali-sensitive bonds. Strong alkali disorients the molecular chains and thus leads to a lower increase than would be expected; if the fibres are stretched after strong alkali treatment there is a great increase in subsequent swelling, owing to an increase in molecular orientation.
J. W. B.

Acetylation of Jute. H. J. Callow. *J. Textile Inst.*, **43**, T 423–T 432 (Aug. 1952).

Dewaxed jute yarn and fabric, unbleached and bleached by NaOCl and H_2O_2, are acetylated by five different methods—acetic anhydride, xylene, and sulphanilic acid; acetic anhydride and pyridine; impregnation with potassium acetate and heating in acetic anhydride; acetic anhydride and sodium acetate; and acetic anhydride alone. Rates of acetylation are followed by KOH hydrolysis and back titration, and colour changes are followed photometrically both after acetylation and after subsequent irradiation by a mercury-vapour lamp. Discoloration can be minimised and even prevented by sufficiently raising the acetyl value; beyond a certain value irradiation causes further bleaching. Acetyl contents of 14–18%, and 16–19%, for dewaxed and bleached jute respectively are required to produce neither discoloration nor bleaching. Fractionation of acetylated material shows that the lignin, *in situ*, acquires acetyl groups more readily than does the cellulose. Tensile and abrasion tests show some loss in dry strength but an increase in wet yarn strength; fabric abrasion resistance is generally reduced.
J. W. B.

Utilisation of Chaff and the like for the Production of Furfuraldehyde as a By-product of Flax Processing. S. Wolf. *Faserforsch. und Textiltech.*, **3**, 225–236 (June 1952).

A survey is made, with numerous references to the literature, of available knowledge concerning the waste products of flax production and the possible ways in which they may be utilised, the technical importance of furfuraldehyde and the chemistry of its formation from vegetable material, and the methods available for the production of furfuraldehyde from flax wastes.
A. E. S.

Removal of Alginate Draw-threads from Wool Half-hose. W. Penn. *Dyer*, **107**, 943 (27 June 1952).

Alginate draw-threads in wool half-hose can be removed by agitating gently in a soln. containing approx. 3 g. Calgon T and 5 g. common salt per litre at 120° F. and then scouring in 1 g. Calgon T per litre.
B. K.

Recent Investigations in Wool Chemistry. J. B. Speakman. *Melliand Textilber.*, **33**, 823–828 (Sept. 1952).

Results of investigations by the author and his collaborators in four different fields of wool chemistry are reported.

In an investigation into the mol. chain wt. of keratin, human hair and three varieties of wool are treated with 1-fluoro-2:4-dinitrobenzene and then hydrolysed. The dinitrophenylamino acids formed are separated chromatographically and estimated colorimetrically, and it is established that in keratin there are seven chain-terminating amino acids—glycine, alanine, valine, serine, threonine, aspartic acid, glutamic acid—present in stoichiometric molar proportions of 8, 2, 4, 2, 8, 1, 2 respectively for the wools and 8, 2, 8, 2, 8, 1, 2 respectively for human hair. The total content of terminal acids is practically the same for all four keratins and corresponds to a mol. chain wt. of ~ 62,000. This and other evidence indicates that keratins vary in structure to a much smaller extent than would be expected from the variability of the S content. Attempts are made to determine the mol. chain wt. by an extension of work already reported (*J.S.D.C.*, **66**, 583–588 (Nov. 1950)), but it is now found that ClO_2 does, in fact, attack certain peptide linkages. Thus, when silk is treated with ClO_2, it becomes at first insol. in cupriethylenediamine soln. (oxidation of the tyrosine residue to a quinone that forms cross-links), but, after further treatment, regains solubility and then has a fluidity, measured by the Garrett–Howitt method, that rises to more than double the original value and then remains const., even when treatment with ClO_2 is continued over a long period. The fluidity figures, determined as for silk, of various ClO_2-treated keratin fibres correspond to average chain weights of 12,600–14,700, but the preparations are very inhomogeneous, extreme fractions of mol. wt. 6,600 and 35,800 being obtained by fractional precipitation of a soln. of treated Lincoln wool (average mol. wt. 12,800).

Data are given of the longitudinal and transverse swelling of wool fibres, both untreated and treated with thioglycollic acid, in formic acid solutions. They are interpreted in terms of the spatial structure of the keratin grid and of the presence in the fibre of less accessible crystalline regions.

A discussion is given of work, already referred to elsewhere (see *J.S.D.C.*, **66**, 657–658 (Dec. 1950); **68**, 278 (July 1952)), on the plasticity of wool.

In an investigation on the mechanism of permanent set, wool fibres, after setting in the stretched condition in boiling borax soln., are treated with 1-fluoro-2:4-dinitrobenzene, hydrolysed, and treated chromatographically for the separation of the dinitrophenyl deriv. of lysine. It is thus shown that the lysine content of wool is reduced from 3·27%, to 2·15–2·53%, during the setting process, which is in agreement with the author's postulate of the participation of lysine in the formation of cross-links, e.g. $CH_2{\cdot}S{\cdot}NH{\cdot}(CH_2)_4$, during setting. It is shown further that, in the hydrolysis both of the original and of the set fibre, the same amount of lysine is formed, which is consistent with the observation that, under the conditions of the hydrolysis, S·NH linkages, e.g. in $NO_2{\cdot}C_6H_3{\cdot}S{\cdot}NH{\cdot}CH_2$, are in fact broken. It is shown also that the failure to detect an increase in the cysteine content of wool after setting may be attributed to the re-formation of cystine during the hydrolysis process necessary for the estimation, for it is found that, when thioamides are boiled with 6 N-HCl in presence of a mercaptan, a disulphide is formed.
A. E. S.

Reversal of Molecular Set in Keratin. J. Sikorski. *Nature*, **170**, 275–276 (16 Aug. 1952).

Wool fibres which have been set in boiling water under tension for 5 min. have their β-form crystallites reconverted to the α-form by treatment in 0·15 N-NaOH, 75% LiBr, or (part reconversion only) 98–100% formic acid solution. Degree of reversal is decreased when setting time is increased; after setting for 5 hr. there is no reconversion in urea and only partial in the above reagents. Cuprammonium hydroxide is, however, found to be very effective. Fibres extended by 50%, and set, no matter for what time, in boiling water are left in the α-form after subsequent immersion in cuprammonium followed by removal of the copper in dilute acid. Similar reconversion is observed in fibres set in steam at 100% extension. Such reconverted fibres still show the normal reversible $\alpha \rightleftarrows \beta$ transformation on stretching and release in water, though somewhat higher extensions are necessary to effect complete change. The fibres can again be set, in which condition they are once more stable to reagents except cuprammonium; the $\alpha \rightarrow$ set $\beta \rightarrow \alpha$ cycle can be followed repeatedly. That the fibres are, in fact, changed is shown by the spontaneous appearance of the β-form on immersion in boiling water—a characteristic of the more labile proteins. It is suggested that some of the linkages responsible for the stability of the α-form are broken during relaxation at high temperatures, their subsequent re-formation being inhibited by cuprammonium, which probably entirely eliminates many of the links.
J. W. B.

Viscosity of Sodium Salt of Polyuronide Hemicellulose from Jute. P. K. Saha. *Nature*, **170**, 281 (16 Aug. 1952).

Viscosity measurements are made on the Na salt of a methylurono acid–xylan complex, which is a jute hemicellulose fraction; as this uronic acid is believed to occur in a chain molecule, it was thought that the Na salt of the complex in aqueous solution might behave as a polyelectrolyte. Results support this conception; the reduced viscosity increases on dilution, and in presence of 0·1 N-NaCl the Na salt behaves as a neutral polymer.

J. W. B.

Degradation of Wool by Heat. M. van Overbeke, G. Mazingue, and P. Despreto. *Bull. Inst. textile France*, (30), 273–288 (Jan. 1952).

Part I discusses the effect of heat on the mechanical properties of the fibres. Hot air results in loss of elasticity of the wool. Comparison of the parameters, temperature and time, shows that the former is the more important. Part II provides results of a detailed chemical study of thermal degradation, whilst the mechanism of the changes produced is discussed in Part III. The principal reaction taking place is—

$$\text{HOOC·CH(NH}_2\text{)·CH}_2\text{·S·S·CH}_2\text{·CH(NH}_2\text{)·COOH} + 2\tfrac{1}{2}\text{O}_2 + \text{H}_2\text{O} \rightarrow 2\text{HOOC·CH(NH}_2\text{)·CH}_2\text{·SO}_3\text{H}$$

Side reactions are also discussed. G. L.

Dissolution of Keratin. J. B. Speakman and J. Menkart. *Bull. Inst. textile France*, (30), 315–323 (Jan. 1952).

The problem has great practical as well as theoretical significance, as regenerated fibres are already produced from protein solutions. The reclamation of wool from mixtures which cannot be efficiently carbonised would be made practicable if a suitable specific solvent for wool could be found. Since the long keratin chains must be preserved by such a solvent, oxidising and reducing agents which attack the disulphide bonds rather than alkaline hydrolytic reagents deserve primary consideration. Finishing processes such as decatising introduce stable side-chain links which make it difficult to dissolve the wool. G. L.

Long-range Elasticity of Hair Keratin. K. H. Meyer, A. J. A. van der Wyk, W. Gonon, and C. Haselbach. *Trans. Faraday Soc.*, **48**, 669–674 (July 1952).

Elastic properties of horse-hair are studied, glycollic acid at pH 4, thioglycollic acid at pH 9, and 70% thioglycollic acid being used as swelling agents in order to reduce the plastic component and emphasise the true elastic component. Thermoplastic behaviour of highly swollen hair from the tail of an Indian elephant is measured under torsion. The stresses observed are mostly plastic, due to cohesive forces, and the elastic stress increases slowly on relaxation, becoming predominant only after a long time. Thermodynamic analysis shows that elastic stress in swollen fibres is similar to that of rubber and elastomers generally. Long-range elasticity in keratin arises from thermal motion of the chain segments in the swollen amorphous fibre. Long-range elasticity is shown neither by unswollen keratin fibres nor by frozen rubber. Two modifications in crystalline keratin are found also in gutta-percha and some polyamides; the transition α- $\rightarrow$ β-keratin does not contribute to long-range elasticity. J. W. B.

Absorption of Water by Hair, and its Dependence on Applied Stress. L. R. G. Treloar. *Trans. Faraday Soc.*, **48**, 567–576 (June 1952).

The report by White and Stam (*Text. Research J.*, **19**, 136 (1949)) that the equilibrium water content of human hair is decreased by application of a tensile stress is not consistent with general thermodynamics, and an argument is presented which predicts an increase in absorption when accompanied by *longitudinal* extension. Experiments carried out on horsehair at 75·5% R.H. show an increase of 0·28% on the dry wt. for a stress of 482 kg./sq. cm., in quantitative agreement with the thermodynamic theory. Data are presented for the dimensional changes which accompany water absorption, and it is calculated that an all-round *lateral* stress of 100 kg./sq. cm. would give a change of 10% out of a total water content of 28%, at 93% R.H. It is suggested that with twisted yarn or cord an overall tension may lead to a *reduction* of water content owing to the greater sensitivity of the fibre to the resultant lateral stress. J. W. B.

Synthetic Fibres. D. Traill. *Bull. Inst. textile France*, (30), 109–130 (Jan. 1952).

A review covering present-day knowledge of the relationship between the physical properties of fibres and their chemical constitution. Production techniques are indicated. G. L.

Synthetic Fibres—Notes on their Properties, Manufacture, and Finishing. E. Pieper. *Textil-Praxis*, **7**, 608–616 (Aug. 1952).

A short review of the production and properties of existing synthetic fibres, particularly Perlon, is followed by notes on the setting and dyeing of Perlon. A. E. S.

Water Sensitivity of Glass Fibre. W. Boheth. *Faserforsch. und Textiltech.*, **3**, 241 (June 1952).

Correction to previous paper, *ibid.*, **3**, 142–146 (April 1952): *J.S.D.C.*, **68**, 324 (Aug. 1952).

PATENTS

Cellulose Derivatives having Affinity for Acid Dyes. Celanese Corpn. of America. *BP* 677,459

Introduction of unsubst. or *C*-subst. morpholino (or morpholinyl) groups into the molecule of cellulose or a cellulose derivative, e.g. by heating an organic sulphonyloxy compound of cellulose or of a cellulose ester or ether with morpholine or a *C*-subst. morpholine until all the sulphonyl groups have been eliminated, yields a product which not only is resistant to gas fading when dyed with 1:4-diaminoanthraquinone dyes but also has affinity for acid dyes. C. O. C.

Linear Polyamides. Monsanto. *BP* 677,516

Linear polyamides of general molecular structure $[\text{CO·R·CO·NH·CH}_x\text{·NH}]_y$ (x = an integer; R = alkylene of > 1 C, which may be interrupted by O or S) can be formed into films and fibres. They are prepared by condensing bifunctional nitriles with formaldehyde. C. O. C.

Solvents for Nylon. Polymer Corpn. *USP* 2,592,616

Nylon, even if normally insoluble in alcohol, can be dissolved in methyl alcohol by heating under pressure in absence of oxygen. C. O. C.

Raising the Safe Ironing Point of Linear Copolymers containing 1:2:4-Triazole and Peptide Links. BrC. *BP* 678,160

Polymers of the type produced by the process of *BP* 627,124 (*J.S.D.C.*, **66**, 80 (1950)), and particularly those produced by condensing a polyaminotriazole with a diamine-stabilised polyamide, have their m.p. raised by treatment with an aldehyde or a di- or poly-*iso*cyanate or a di- or poly-urethan. C. O. C.

Solutions of Acrylonitrile Polymers. American Viscose Corpn. *BP* 677,738

A mixture of nitromethane (90–98% by vol.) and formamide (10–2) yields clear, colourless solutions, of up to 25% strength, which do not show gels or undissolved particles on microscopic examination, remain stable on prolonged storing, and can be dry-spun at low temperatures. C. O. C.

Solvents for Acrylonitrile 2-Methylallyl Alcohol Polymers. Eastman Kodak Co. *USP* 2,591,670

Acrylonitrile–2-methallyl alcohol polymers are soluble in volatile solvents, e.g. acetone or acetonitrile, or mixtures of such solvents with water or other materials. The solutions so obtained can be used to produce filaments by either dry or wet spinning. C. O. C.

Rubber Polymer Sulphur Dioxide Filaments. Shell Development Co. *USP* 2,591,254

Readily woven filaments are obtained by extruding a solution of rubber or other high-mol.wt. unsat. rubbery polymer, which solution contains an oxide of chlorine (0·05–5·0% on wt. of polymer) into a coagulating bath containing SO_2. C. O. C.

Asbestos Fibre Recovery. Johnson's Co. *BP* 677,780

Asbestos-containing ore is made into an aqueous slurry of 1–15% solids and not < 2·5% asbestos content, this last being at least 40% in the fluffed state. The slurry, which must screen at 6–16 mesh with respect to the solids, is washed with a jet of water over a shaking table, the

action of which separates a fibre concentrate from the sand residue. Drying of large quantities of extraneous material is avoided, low grade fibre is recovered, and the product is comparatively free from dust and iron. J. W. B.

Acetylene in Chemical Industry (III p. 463).
Structure of Proteins (XII p. 481).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Dipole Moments of Tervalent Metallic Soaps in Benzene. B. C. Bannerjee and S. R. Palit. *J. Indian Chem. Soc.*, **29**, 175–182 (March 1952).

The dipole moments of Cr, Al, and Fe oleates, Fe laurate, and Fe stearate have been calculated from dielectric measurements using the Debye equation, the order for the oleates being found to be Cr > Al > Fe. Addition of a small quantity of alcohol to a benzene solution of Al and Fe oleates, which is known to have a great effect on the viscosity of the solution, gave a large increase in the dipole moment, which then fell sharply on the addition of more alcohol. This is due to micellar breakdown and solvation of the soap molecules by alcohol, followed by coagulation on the addition of more alcohol. C. H. R.

Deposition of Soiling Matter from Detergent Systems. I—Aqueous Systems. II—Non-aqueous Systems. R. E. Wagg. *J. Textile Inst.*, **43**, T 325–T 330; T 331–T 337 (July 1952).

I—Bleached cotton fabric is agitated with different detergent solutions containing a constant amount of added soiling matter (ilmenite), and the amount of dirt deposited on the material is measured photometrically. None of the synthetic detergents examined is as good as soap (Na oleate) in preventing deposition, but their properties are improved by adding Na carboxymethyl cellulose (C.M.C.). Addition of Na_2CO_3, and to a lesser extent NaCl, cause more deposition than Na metasilicate; addition of C.M.C. partly overcomes the deposition effect of Na_2CO_3. Even very low concn. (about 0·0015%) of C.M.C., which are more acceptable economically, have some effect in reducing deposition, though the reflectance–concentration curve does not flatten until a concn. of about 0·01% is reached.

II—Scoured unbleached knitted woollen fabric is conditioned at different R.H. and shaken in solvents (trichloroethylene and white spirit) containing various additions—superfatted soaps, synthetic detergents, ethyl cellulose, etc.—to which a standard soiling matter has been added. Light reflectance is measured photometrically before and after the procedure. With wool preconditioned at 0, 70%, and 100% R.H. at 25° C. more deposition occurs from white spirit alone than for trichloroethylene alone. The varying effect of added compounds is influenced by the condition of the wool, but in some instances coagulation of additive and dirt occurred and could not be correlated with the final deposition. J. W. B.

Rapid Continuous Bleach for Low-grade Cotton Goods. B. S. Voronkov and R. M. Shchegoleva. *Tekstil. prom.*, **12**, 29–32 (May 1952).

The continuous bleach employed at the Krasnaya Talka printworks, which is satisfactory for medium-weight cotton fabrics of normal quality, is not effective for heavier fabrics containing low-grade cotton, particularly with respect to the removal of motes. In this bleach, the goods are treated in a continuous apparatus in a soln. containing per litre—15–19 g. NaOH, 5 g. Kontakt (surface-active agent), 5 g. sodium silicate, and 8 g. sodium sulphite; the bleach is not described in detail. It is now found that low-grade cottons can be treated satisfactorily by giving prescouring treatments in alkali and in hypochlorite. The grey fabric is impregnated in hot spent lye (~6 g. NaOH per litre) from the continuous scour and is then passed to a piler (1 hr. passage). It is washed with cold water, impregnated with calcium hypochlorite soln. (active Cl—1·1–1·2 g./litre), given a 1-hr. passage in a piler, and again washed. It is then impregnated in the hot scouring soln. (~17 g. NaOH and 2 g. Kontakt per litre) and passed immediately into the continuous kier. The further stages of washing, hypochlorite treatment, and souring are not given in detail. The finished fabric is compared with material from the usual continuous bleach and from the static kier bleach as regards whiteness, absorbency, nitrogen content, wax content, cuprammonium viscosity, and freedom from motes; it is of high quality in all respects. The preliminary treatment in alkali, as well as increasing the effectiveness of the scour, prevents the evolution of obnoxious chloramines during the succeeding hypochlorite treatment. A. E. S.

Action of Hydrogen Peroxide on Jute—I and II. D. B. Das, M. K. Mitra, and J. F. Wareham. *J. Textile Inst.*, **43**, T 443–T 449; T 449–T 453 (Aug. 1952).

I—Jute is bleached with different concentrations of H_2O_2 and the weight changes, changes in cuprammonium fluidity and Cu number of α-cellulose, and contents of α-cellulose and of lignin as determined by H_2SO_4 and chlorite processes are studied. It is suggested that the chlorite process should be used with caution when the jute has received chemical pretreatment, as substances (e.g. hemicellulose) other than lignin are also removed. Up to 1-vol. H_2O_2 (liquor ratio 20 : 1) in presence of alkali at 75–85°C., α-cellulose is unattacked, but 4·2% of hemicellulose goes into solution along with the lignin. Above 1 vol., α-cellulose is attacked, and more lignin, but no more hemicellulose, is lost. At 20-vol. concn. and 100 : 1 ratio it is possible to remove only 4·6% lignin and 4·5% hemicellulose. It is suggested that part of the lignin or hemicellulose is either differently constituted or located from the remainder.

II—Water absorption at different R.H. and wet and dry tensile properties of jute treated with different concentrations of H_2O_2 under controlled time, temperature, and liquor ratio are investigated. Moisture absorption is roughly independent of H_2O_2 treatment over a range of R.H. (0–92%), and below 2-vol. H_2O_2 concn. the dry strength is increased and wet strength greatly reduced. This supports the suggestion that lignin lies between the disorganised molecular chains, preventing higher crystallinity and leading to a greater hygroscopicity than in cotton. Oxidation by H_2O_2 breaks the lignin–cellulose or hemicellulose linkage and results in new hydrogen bonds, which are effective in the dry state but not as effective as the old ones when saturated; thus there is a lower absorption of moisture at R.H. 9·4% and a greatly increased one at 100%. J. W. B.

Decolorisation of Dye Solutions by Ultrasonic Waves. II—Triphenylmethane Dyes. T. Azami and T. Hinohara. *J. Chem. Soc. Japan, Pure Chem. Sects.*, **72**, 515–517 (1951): *Chem. Abs.*, **46**, 6479 (25 July 1952).

Aqueous solutions (0·003%) of Crystal Violet, Malachite Green, Brilliant Green, and Fuchsin were bleached by ultrasonic irradiation. Rosolic acid, phenolphthalein, and aluminon were not decolorised. The bleaching action is due to the oxidative action of oxygen which has been activated, in particular, at the interface between the solution and the air. C. O. C.

PATENT

Cleaning Furs. N. Root. *USP* 2,591,663

The furs are drummed with sponge-cloth bags of different sizes loosely filled with sawdust or other cleansing material. The weave of the sponge cloth must allow solvent to be expressed through it but prevent the sawdust from sifting through it. This avoids the sawdust getting into the furs, and is therefore especially suitable for treating "Mouton" (shorn-lamb), Persian lamb, etc. C. O. C.

Washing off Printed Naphtholated Fabrics (IX p. 478).
Estimation of the Whiteness of Optically Whitened Textiles (XIV p. 482).

VIII—DYEING

Potentiometric Control in Dyeing. J. Rath. *Melliand Textilber.*, **33**, 862–864 (Sept. 1952).

The use in the U.S.A. of the "Marben" redox potentiometric control in vat dyeing (see J.S.D.C., **68**, 369 (Sept. 1952)), particularly in continuous processes, enables more regular results to be obtained and conduces to the avoidance of wasteful additions of hydrosulphite. Over-reduction of Indanthren Blue BC can be prevented by adding sodium nitrite and hydroxylamine to the dyebath; a high processing temp., e.g. 91°C., can then be used, satisfactory reduction being obtained at a redox potential of 260 mv. less than that normally required. A. E. S.

Dyeing of Textile Fibres at High Temperature. L. Drijvers. *Amer. Dyestuff Rep.*, **41**, 533–539 (1 Sept. 1952).

English translation of paper, see J.S.D.C., **68**, 325 (Aug. 1952).

Dyeing in the Blended Fibre Hosiery Industry, with some Observations on Finishing. C. S. Groves and C. F. Ward. *J. Textile Inst.*, **43**, P 626–P 647 (Aug. 1952).

After brief discussion of the dangers inherent in finishing blended fibres, e.g. severe bleaching which may be necessary for one component will damage the other, the processes of scouring, bleaching, and dyeing are reviewed at length. Attention is paid to three groups of blends—(1) Wool plus cotton, regenerated cellulose, nylon, Ardil, Vicara, shrink-resistant wool, acetate rayon, and Rayolanda; (2) cotton plus Rayolanda, regenerated cellulose plus Rayolanda, and cotton plus viscose, cuprammonium, and acetate rayon; (3) miscellaneous synthetic polymer fibres. Particular attention is paid to dyeing wool–nylon mixtures. Shrinkage properties, raising, and process control are also discussed. J. W. B.

Modifying Dyeing by Sound Waves—III. H. Rath and H. Merk. *Melliand Textilber.*, **33**, 859–861 (Sept. 1952).

Experiments similar to those already reported for audible sound (cf. J.S.D.C., **68**, 227–228 (June 1952)) are carried out with ultrasonic waves of 22–175 kilocycles/sec. and 1–3 watt/sq. cm. Exact comparison with the results of the previous experiments is not possible, but the accelerating effect on the dyeing rate at 1 watt/sq. cm. is approx. the same as that obtained with audible sound at 0·5 watt/sq. cm. The effect is enhanced by increase in intensity, but is substantially independent of the frequency over the range investigated. Vat dyeings obtained in presence of ultrasonic waves are inferior in solidity. A. E. S.

Dyeing with Azo Dyes. H. Gerstner. *Faserforsch. und Textiltech.*, **2**, 282–288 (July 1951): *J. Textile Inst.*, **34**, A 529 (July 1952).

This is a review of the literature concerning the nature of the dyeing action of azo dyes. There are fifty references. C. J. W. H.

Problem of Rubbing Fastness in Azoic Dyeing. W. Wüterich. *Melliand Textilber.*, **33**, 743–747 (Aug. 1952).

A discussion of the conditions that must be observed at each stage of azoic dyeing in order to ensure max. fastness to rubbing. A. E. S.

Theory and Practice of the Application of 4-Methoxy-4′-diazodiphenylamine [Variamine Blue B Salt] in Dyeing. V. A. Blinov. *Eighth Aniline Dye Conference (U.S.S.R.)**, 79–87.

The wet development of Variamine Blue on fabric is often accompanied by a poor yield and bad penetration. The conditions under which coupling proceeds are studied by observing the development of the dye on fabric and the behaviour of the coupling components *in vitro*, e.g. when brought together in soln. under various conditions of pH and when titrated potentiometrically with acids or alkalis. Titration and coupling of the diazo component are carried out also in presence of the octadecanol–ethylene oxide condensate Emulphor O, which facilitates examination of alkaline solutions by maintaining the non-coupling diazo-hydroxide in colloidal soln. In alkaline soln. the diazonium ion is converted into its active form (following the hypothesis, due to B. A. Poray-Koshits and Grachev, of its existence as a pair of tautomeric cations (see *Chem. Abs.*, **41**, 1215 (1947)), and the rate of coupling *in vitro* rises progressively with pH; but at pH $>$ 10·5, the active cation is rapidly converted into the diazo-hydroxide and, under the conditions of development on fabric, no appreciable coupling can occur. At pH $<$ 8, the other component (Azotol A, i.e. Naphtol AS) is in its inactive, undissociated form, and coupling is inhibited. Consequently, coupling is confined to the pH range 8–10·5. The pure diazonium chloride can be estimated in soln. by adding an excess of alkali, allowing to stand for 30 min. so that conversion into insol. diazo-hydroxide may be complete, and back-titrating potentiometrically with acid until the pH falls sharply. A method is recommended for the more complete utilisation of the components in dyeing. After passing the fabric through the diazo soln., it is given a passage of at least 90 sec. before squeezing, and is then passed into a soln. of HCl in order to reconvert diazo cpd. that became inactive under the alkaline coupling conditions into diazonium salt; the fabric is then passed into a buffer soln. of pH 11, when further coupling occurs. A. E. S.

* See J.S.D.C., **68**, 460 (Nov. 1952).

Investigations on Colour Development. III—Behaviour of Diethyl-*p*-phenylenediamine in Aqueous Solution containing Hydroxylamine towards Atmospheric Oxygen. IV—Effect of Copper Ion and of Calcium-sequestering Agents on Air Oxidation of Colour Developers containing Hydroxylamine. K. Meyer and W. Brune. *Z. wiss. Phot.*, **46**, 169–174; 174–178 (1951): *Chem. Abs.*, **46**, 7445 (25 Aug. 1952).

III—When a solution containing diethyl-*p*-phenylenediamine (I), Na_2SO_3, and K_2CO_3 is oxidised by air, the concn. of I and Na_2SO_3 decrease linearly with increasing duration of aeration. NH_2OH retards loss of I but increases rate of loss of Na_2SO_3. One mol. of NH_2OH is consumed for each 2 mol. of Na_2SO_3 lost. Apparently an addition or complex compound of sulphite and NH_2OH is the effective preservative. No significant change in concn. of I occurs during aeration when NH_2OH is present until the Na_2SO_3 concn. falls to a low value.

IV—$CuSO_4$ markedly increases the rate of oxidation of I by air. Trilon B (II) nullifies the action of Cu but causes increase in rate of oxidation in absence of Cu. Rate of loss of Na_2SO_3 is increased only slightly by II, but rate of loss of NH_2OH is greatly increased, which accounts for the increase in rate of oxidation of I. II markedly increases rate of decomposition of NH_2OH in alkaline soln., and this accounts for the increased loss. Rate of decomposition of NH_2OH in alkaline solution containing II is increased by presence of O_2. Calgon used as a Ca-sequestering agent does not accelerate decomposition of NH_2OH but offers no protection against Cu. C. O. C.

Polarographic Reduction of Crystal Violet, Brilliant Green, Malachite Green, and Auramine. R. C. Kaye and H. I. Stonehill. *J.C.S.*, 3231–3239 (Aug. 1952).

All four dyes are reducible in aqueous solution at the dropping Hg electrode, giving polarograms distorted by dye adsorption on the Hg. Elimination of the latter, by addition of ethanol, furnishes normal polarograms which exhibit two well separated one-electron steps, indicating formation at the first step of a semiquinone free radical of apparently very great stability. The halfwave potential of the first reduction step appears to be independent of pH around pH 7, so that, as with the aminoacridines (cf. *J.C.S.*, 2638 (1951): J.S.D.C., **68**, 46 (Jan. 1952)), semiquinone formation involves the uptake of one electron and no proton. H. H. H.

Polarographic Reduction of Pyridine, Quinoline, and Phenazine. R. C. Kaye and H. I. Stonehill. *J.C.S.*, 3240–3243 (Aug. 1952).

Pyridine, quinoline, and phenazine do not form highly stable semiquinones on polarographic reduction at pH $\sim$ 7, but quinoline and phenazine form relatively unstable ones. H. H. H.

Polarographic Reduction of some Natural and Artificial Hydrogen-carriers in Bacterial Enzyme Systems. R. C. Kaye and H. I. Stonehill. *J.C.S.*, 3244–3247 (Aug. 1952).

Cozymase, riboflavin, methylene blue, Capri Blue, and cresyl blue, in aq. soln. at pH 7·38, and Nile Blue in 50% ethanol at pH 8·29, undergo polarographic reduction at 25°c. in two overlapping one-electron stages, with the intermediate formation of semiquinones whose stability is low compared with those from several aminoacridines and di- and tri-phenylmethane dyes. H. H. H.

Measurement and Practical Significance of the Redox Potentials of Vat Dyes. R. H. Peters and W. J. Marshall. *Bull. Inst. textile France*, (30), 415–438 (Jan. 1952).

The intrinsic characteristics of vat dyes which have a bearing on their technical applications are their redox potentials and their rates of reduction. The speed of reduction is a function of temperature, concentration of

$Na_2S_2O_4$, crystalline form, and particle size. The predominant factor determining reduction rate is individual to the dye itself, particle size per se being only of secondary significance. G. L.

Redox Potentials of some Polyhydroxyanthraquinones and Vat Dyes. H. W. Meyer and W. D. Treadwell. *Helv. Chim. Acta*, **35**, 1444–1460 (Aug. 1952).

The redox potentials of thioindigodisulphonic acid, indigodisulphonic acid, anthraquinone-β-sulphonic acid, 1:5- and 1:8-dihydroxyanthraquinone-3:6-disulphonic acids, alizarin-6-sulphonic acid, cresyl blue, Lauth's violet, thiazine blue, methylene blue, new methylene blue, Safranine O, and neutral red have been measured as a function of acidity throughout a range extending from strongly acid (pH 0) to alkaline (pH 10). The results are given in tables and acidity potential curves. Semiquinone formation has been established for the cases of cresyl blue and 1:8-dihydroxyanthraquinone. H. H. H.

Influence of Acidity on the Absorption Spectrum of some Vat Dyes and its Relationship to the Redox Potential. H. W. Meyer and W. D. Treadwell. *Helv. Chim. Acta*, **35**, 1460–1470 (Aug. 1952).

The absorption spectra of the dyes dealt with in the previous abstract have been measured in acid and in some cases in alkaline solution, and displacements caused by salt formation are discussed. H. H. H.

Dyeing of Acetate Rayon Yarn. G. Bertolina. *Rayonne, Fibres synth.*, **8**, (1), 11–26 (1952); *J. Textile Inst.*, **43**, A 527 (July 1952).

Dyeing procedures used to dye acetate rayon in Europe (most particularly in Italy, France, and England) are surveyed. This comprehensive survey includes—(1) details of methods using disperse, soluble, "diazo", vat, soluble vat, mordant, direct cotton, acid, and basic dyes; (2) lists of dyes found to be reliable; and (3) indications of whether the procedures are suitable for dyeing skeins, cakes, or rayon staple. Rayon production figures for the U.S.A. show the increasing popularity of acetate rayon there. Acetate rayon dyeing in the U.S.A. is about the same price as viscose rayon dyeing, whilst in Italy it is about twice as expensive. C. J. W. H.

Protection by Protein Agents in the Dyeing of Wool. M. van Overbeke, G. Mazingue, and R. Laloy. *Bull. trimestriel, Chambre Commerce Roubaix*, (12), 9–25 (1951); *J. Textile Inst.*, **43**, A 530 (July 1952).

The deterioration of wool dyed in hot concentrated acid or alkaline baths is counteracted by the use of protective protein agents. Tests were carried out without and then with protective agents of differing molecular weight obtained from gelatin, and results showed that optimum protection was given by those of mol. wt. ~ 2700. The concentration of the agent had little effect at pH 4–7, but in more acid or alkaline media greater concentrations gave a better protective effect. Physical tests showed that the agents do not affect the tenacity of the fibres, but make them more plastic. C. J. W. H.

Dyeing of Perlon and Nylon Textiles. W. Ender and F. Wenger. *Textil-Praxis*, **7**, 630–635 (Aug. 1952).

A discussion is given of the dyeing of Perlon and nylon textiles with acid, chrome, vat, and disperse dyes, including chromium-complex dyes of the acid and disperse classes (the latter are available as Vialon Fast (Basf) dyes). The properties of the dyeings and their suitability for various classes of work are discussed. A. E. S.

PATENTS

Hydroxyalkyldiamines — Dyebath Assistants for Difficultly Soluble Metal-complex Direct Cotton Azo Dyes. Ciba. *BP* 676,584

Difficultly soluble Co, Ni, and Cu complexes of o-hydroxycarboxy-, *oo'*-dihydroxy-, and *o*-carboxy-*o'*-hydroxy-azo dyes may be successfully dyed on cellulosic fibres in presence of an aliphatic diamine—

$$C_nH_{2n}(NH\text{-}CR^1R^2\text{-}CH_2OH)_2$$

(R^1 = H, CH_2OH, or Alk of < 4 C; R^2 = H or CH_3; n < 5, and preferably = 2). Or a mixture of the diamine, a suitable metallic salt, and the unmetallised dye may be used in the dyebath. Thus, all parts being by weight, 1:2-bis-(β-hydroxyethylamino)ethane (1) and the Cu complex (1) of the trisazo dye 5-aminosalicylic acid $\xrightarrow{1}$ 1-naphthylamine-7-sulphonic acid $\xrightarrow{2}$ α-naphthylamine $\xrightarrow{3}$ γ acid are boiled with water (200). The solution so produced is diluted (to 3000), soda ash (2) is added, and then cotton (100) at 60°C. After boiling for 5 min. Glauber's salt (20) is added, and after 20 min. a further addition (20). A blue of excellent fastness to light and washing results. E. S.

Single-bath Process for Producing Coppered Azo Dyes on Cellulosic Fibres. Ciba. *BP* 676,647

Dis- and poly-azo direct cotton dyes which contain at least one *oo'*-dihydroxy- or *o*-carboxy-*o'*-hydroxy-azo group, and which yield difficultly soluble copper complexes, may be applied from a bath containing ammonia, or an aliphatic base, and a compound containing copper in a complex anion. Thus, all parts being by weight, the disazo dye (1) 4:4'-diaminodiphenyl-3:3'-dicarboxylic acid $\rightarrow$ (3-methyl-1-*p*-sulphophenyl-5-pyrazolone)$_2$ is boiled with water (100) and diluted with water (2900) at ca. 60°c., and then soda ash (2) is added. A solution (60) prepared from $CuSO_4.5H_2O$ (50), sodium citrate (50), and monoethanolamine (100) diluted with water (to 1000) is added to the dye solution, and then cotton (100) is entered and the bath brought to 100°c. in 30 min. Boiling is continued for 45 min., an addition of Glauber's salt (20) being made after 5 min. and again after 20 min. An orange of good fastness to light and washing is obtained. E. S.

Dyeing Wool with High-affinity Colloidal Dyes and Diammonium Phosphate. Pacific Mills. *USP* 2,590,847

Use of diammonium phosphate (0·1–0·5% on the wt. of liquor) as the dyeing assistant enables the bath to be at pH approx. 7·1 at the start, 6·8 at the boil, and 6·5 after 2 hr. boiling, and this enables well levelled dyeings to be obtained with difficultly levelling neutral-dyeing acid and metachrome dyes. C. O. C.

Aftercoppering on Nylon. ICI. *BP* 678,106

Copperable dyes can be satisfactorily aftercoppered on nylon by cuprous salts in acid solution in presence of an alkali-metal halide. C. O. C.

Studies in Vat Dyes. IV—Preparation and Dyeing Properties of Di-imines from 2:7-Dibromophenanthraquinone (IV p. 467).
Capillary Phenomena in Assemblies of Fibres (VI p. 471).
Treating Loose Fibres (X p. 480).

IX—PRINTING

Lytax Textiloscope. H. Eber. *Melliand Textilber.*, **33**, 764–766 (Aug. 1952).

The Lytax Textiloscope is an optical device for use on roller printing machines. It provides the printer with a stationary image of the pattern, which is formed not on the simple stroboscope principle, but by maintaining the group of repeats under view for practically the whole of its passage through the field of the instrument and then switching over within a very short space of time to a coincident view of the next group of repeats. The principle of operation is described. The instrument can be used for repeats of up to 60 cm. and for fabric speeds of up to 100 m./min. A. E. S.

Washing off Printed Naphtholated Fabrics. S. A. Plaksin and A. S. Makarova. *Tekstil. prom.*, **12**, 33–34 (May 1952).

Comparative data are given of the degree of removal of Azotol (Naphtol AS) from a closely woven naphtholated fabric by means of water and of alkaline solutions. The leaching effect of water is increased by softening, further increased by small additions of NaOH (2 g./litre), and still further increased by the addition of NaOH and a surface-active agent; it increases rapidly with rise in temp. and is improved, particularly at low temp., by increasing the rate of circulation of liquor through the fabric. A. E. S.

Javan Batik Industry. W. Kertscher. *Melliand Textilber.*, **33**, 161–163 (Feb.), 222–226 (March), 435–438 (May), 634–636 (July), 868–870 (Sept. 1952).

A comprehensive survey, with many illustrations and recipes, is given of the processes employed in the production of Javan batik, using both natural and synthetic dyes. A. E. S.

Printing of Polyamide Fabrics. C. Kuch. *Textil-Praxis*, **7**, 638–641 (Aug. 1952).

The use of various types of dye for the printing of polyamide fabrics is discussed. The more usual thickenings have rather poor printing properties on these fabrics, but polyvinyl alcohol soln. is very satisfactory, and a brand of this agent, Vinarol 8T, is now available that is compatible with small amounts of alkali. Patterns of direct prints with acid dyes and of a seersucker effect, produced by printing a soln. of resorcinol in stripes, accompany the text. A. E. S.

Colour Photography. R. B. Collins and C. H. Giles. J.S.D.C., **68**, 421–457 (Nov. 1952).

Yield of Dyes in Colour Development. S. A. Bongard, A. N. Iordansky, and V. S. Cheltsov. *Doklady Akad. Nauk S.S.S.R.*, **84**, 81–84 (1 May 1952).

Work on the ratio of the amounts of dye and silver formed during colour development (*cf.* J.S.D.C., **67**, 350 (Sept. 1951)) is extended to three other types of dye in commercial use. The emulsion components used are derived from an aroylacetanilide (yellow), from pyrazolone (red), and from 1:4-naphtholsulphonic acid (blue), the developer being *p*-diethylaminoaniline in each case. The yields are 84%, 76%, and 94% respectively of the theoretical amount (0·25, 0·25, and 0·5 molecule of dye per Ag atom). As predicted from theoretical considerations, the yield is const. throughout the course of the development process, and does not depend on the duration of exposure or development, or on the grain size of the emulsion. A. E. S.

Flexichrome Process. F. W. Coppin and J. J. Hook. *Phot. J.*, **91 A**, 173–174 (1951): *Chem. Abs.*, **46**, 6531 (25 July 1952).

Flexichrome film, which is coated on a stripping base, is exposed through the base, and then developed in a tanning developer which hardens the gelatin in proportion to the amount of Ag produced. The unhardened gelatin is removed in hot water, the Ag removed by a bleach, and the hardened gelatin relief-dyed with a black dye which is absorbed according to the thickness of the gelatin. The black image together with a thin carrier is stripped from the base in 2% acetic acid at 38°c. and transferred to a dye-transfer paper. When dry, the print is coloured by special dyes applied with a brush. The dyes replace the black, and will replace one another in the order in which they are applied. C. O. C.

PATENTS

Printing with Direct Cotton Dyes. Ciba. *BP* 677,388

Prints which are fast to light and washing can be produced by using the difficultly soluble metal complex of a direct cotton dye in a paste containing a compound of formula $C_nH_{2n}(NH{\cdot}CR^1R^2{\cdot}CH_2OH)_2$ (R^1 = H, Alk of < 4 C, or CH_2OH; R^2 = H or CH_3; $n < 5$), *e.g.* 1:2-bis(β-hydroxyethylamino)ethane. C. O. C.

Treatment of Back Grey in Screen Printing. E. Owren and E. Karolczuk. *USP* 2,590,643

The surface of the back grey next to the material to be printed is treated with a repellent for the printing colour in use, the repellent being preferably applied as a fine spray between changes of colour and/or pattern, so as to seal in completely dyes previously deposited on the surface of the back grey and thus enable it to be used repeatedly without being washed. C. O. C.

Studies in the Surface Tension Measurement of some Colloidal Solutions (III p. 463).

Investigations on Colour Development. III—Behaviour of Diethyl-*p*-phenylenediamine in Aqueous Solution containing Hydroxylamine towards Atmospheric Oxygen. IV—Effect of Copper Ion and of Calcium-sequestering Agents on Air Oxidation of Colour Developers containing Hydroxylamine (VIII p. 477).

X—SIZING AND FINISHING

Sizing of Textile Fibres—General Considerations. A. Parisot. *Bull. Inst. textile France*, (30), 387–404 (Jan. 1952).

The size should maintain the fibres in an adequately damp condition in order to prevent the effects of static electricity. The fatty phase should be of low viscosity, and it should be capable of acting as a lubricant when spread in thin layers. The fatty matter must be free from any tendency to oxidation, and an antioxidant should be added if necessary. If the size is to be applied in the form of an emulsion, the aqueous phase should have good wetting power. G. L.

Finishing of Fabrics containing Wool and Man-made Fibres. C. S. Whewell, P. Barrett, and J. Senior. *J. Textile Inst.*, **43**, P 648–P 660 (Aug. 1952).

Experiments are carried out to assess the changes in dimensions and appearance of several blended cloths of similar constructions after scouring, milling, and raising. Blends include wool plus Vinyon, nylon, Fibrocetа, Fibro, Fibrolane, and Rayolandia. Rate of milling and limiting milling shrinkages are measured at different pH values, and the effect of chlorination in reducing shrinkage is also investigated. Measurements of pile thickness of wet- and dry-raised fabrics are accompanied by analyses of the % rayon in the pile as compared with that in the original unraised cloth. In conclusion it is suggested that, in producing a typical wool-type fabric with orthodox designs and finishes, it is not desirable to incorporate > 30% rayon, or design and finishing problems will arise. J. W. B.

Raising of Textile Fabrics. C. S. Whewell. *Bull. Inst. textile France*, (30), 439–450 (Jan. 1952).

Scientific methods employed for the measurement of the effectiveness of treatment are carefully discussed. The importance of the weaving construction in determining the ease of raising is demonstrated. Increased moisture content, swelling agents such as weak acids, reducing agents, and lubricants, all facilitate the production of pile on wool. The raisability of fabrics containing rayon or nylon is also improved by the presence of acids and lubricants. A fabric with a cotton warp and possessing > 25% of nylon staple or Fibro in the weft can produce a material having an appearance and handle similar to wool without actually containing wool. G. L.

Prevention and Eradication of Wool Pests with continuously Dispersed Insecticides. A. H. Tocrydlowski and S. Moore. *J. Econ. Entomol.*, **44**, 1016–1017 (1951): *J. Textile Inst.*, **43**, A 534 (July 1952).

Preliminary investigations are reported on the effect of continuously vaporised Lindane on clothes moth and carpet beetle in raw and processed wool stored in rooms of various cubic capacity. The webbing clothes moth was eliminated by the dispersal of Lindane at a rate of 1 g. in 24 hr. in rooms of capacity 15,000–36,000 cu ft., but not in a volume of 48,000 cu. ft. Carpet beetle species *Anthrenus scrophulariae* were eliminated. Adults of *Attagenus piceus* survived but apparently failed to reproduce. C. J. W. H.

Application of some Synthetic Resins in the Textile Industry. A. Landolt. *J. Textile Inst.*, **43**, P 331–P 340 (July 1952).

An account of thermoplastic synthetic resins deals briefly with copolymers, polymerisation technique, and polymer hardness; thermosetting resins from melamine- and urea–formaldehyde are considered more fully, and application techniques described in detail, including the production of a chintz finish with melamine–formaldehyde. J. W. B.

Chloropropanols for Dimensional Stabilisation of Cellulosic Fabrics. C. Musset. *Ind. textile*, **69**, 103–107 (1952): *Chem. Abs.*, **46**, 4393 (10 July 1952).

Cellulosic fabrics can be dimensionally stabilised by many aliphatic compounds containing 3–6 C, 2 Hal, and 1 OH. Drawbacks of the usual stabilising treatments, viz. thermosetting resins, aldehydes, and mechanical methods, are discussed. Use of 1:3-dichloro-2-propanol and 2:3-dichloro-1-propanol is advised, stabilisation being due to polymeric $O{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}O$ cross-linkages between approx. each 300th glucose radical. Treatment is carried out in presence of NaOH and alkali-metal halides at 80–90°c. Detailed recipes are given for stabilising viscose rayon and cotton fabrics. The process leaves a residual shrinkage of 0–2·2% and has negligible effect on both dry and wet breaking load and elongation. C. O. C.

Fireproofing of Cellulosic Fibres by means of Aminoalkyl Sulphamates. J. B. Puig. *Bull. Inst. textile France*, (30), 405–414 (Jan. 1952).

A review of theories of the action of fireproofing agents

is followed by practical details for the application of aminoalkyl sulphamates. When used in conjunction with auxiliaries, a 20–25% impregnation is claimed to be effective without impairing either the handle of the fabric or the effects of previous dyeing or finishing processes. G. L.

Elimination of Water from Textiles. J. M. Preston. *Bull. Inst. textile France*, (30), 467–475 (Jan. 1952).

A critical discussion of the fundamental principles involved in the drying of textile materials. G. L.

Water Repellency. I—Mechanical Theory of the Wettability of a Fabric under the Action of Rain. G. Serchi and P. Chini. *Chimica* (Milan), **7**, 43–55 (1952). *Chem. Abs.*, **46**, 7774 (25 Aug. 1952).

Review. The optimum conditions for obtaining water repellency of fabrics are discussed. C. O. C.

PATENTS

Treating Loose Fibres. Canadian Celanese. *BP* 677,573

The fibres are suspended in the treating liquor and carried by it through a totally enclosed passage kept full of the liquor, the length of the passage and the rate of flow being such that treatment is completed as the liquor carrying the fibres leaves the passage. The process is very suitable for saponifying acetate rayon staple fibre.

BP 677,597

If the fibre content of the suspension is kept at < 2% by weight then the process can be used for dyeing. C. O. C.

Finishing Wool Fabrics. F. Kostring. *BP* 678,585

In finishing wool goods, decatising and heavy pressing can be omitted if the wet fabric is drawn over one or more smooth hot surfaces while being kept damp. Any excess moisture is then removed, e.g. by passing over a suction slit, and the fabric passed without tension over a large hot polished drying cylinder. This imparts smoothness and glaze. If necessary the fabric is cropped, mended, and raised. It is then damped by application of heat and moisture, and fed in absence of tension into a freezing chamber at — 1 to — 20°C. Excess moisture is then removed, and after the fabric has been conditioned it is given successive light flat pressings, preferably between pressboards at low pressure, and put in a chamber at 70°C. and then in one at slightly above 0°C. This results in a full, soft handle and a rich, soft, natural lustre. C. O. C.

Introducing Hollow Spaces into Viscose Rayon. Feldmühle. *BP* 678,155

Viscose rayon is treated successively in either order with aq. H_2O_2 and an aq. soln. which liberates gaseous O_2 by reacting with or acting as a decomposition catalyst for H_2O_2. This transforms the rayon into a porous spongy form having better thermal insulating powers, softer and fuller handle, wavy, curly or grained surface, reduced lustre, and improved permeability to dyes and finishes. This transformation is not accompanied by appreciable decrease in strength. C. O. C.

Crease-resisting of Natural or Regenerated Cellulose Materials. O. J. Meijer's Dextrinefabrieken. *BP* 676,419

The material is impregnated with an aqueous solution of an aldehyde and a nitrogenous reaction product of a polyhydric aldehydo- or keto-alcohol of > 3 C with either (*a*) a compound of formula $NH_2CX{\cdot}NH{\cdot}R$ (R = H, Alk. Ar, or aralkyl; X = O, S, or NH), e.g. glucose ureide, or (*b*) a melamine, and sufficient acid or acid-forming compound to keep the pH of the material upon subsequent drying and heating not > 2. C. O. C.

Protecting Wool against Textile Pests. FBy. *BP* 677,293

Wool is made highly resistant to textile pests by adding compounds containing the $SO_2{\cdot}NH$ group, e.g. chloromethanesulphontrichloroanilide, to the alkaline milling or scouring liquors with which it is treated. C. O. C.

Improving the Water Sensitivity of Nylon. DuP. *USP* 2,592,473

Nylon, fibre or film, is treated with the *N*-carboxyanhydride of an α-amino acid of 1–7 C, preferably substituted in the ring N, e.g. by Alk of 1–7 C, for 15 min.–48 hr. at not > 160°C. Thus nylon yarn refluxed for 24 hr. with its own weight of the *N* carboxyanhydride of sarcosine, after being freed from chlorobenzene, was stiffer but was indistinguishable microscopically from untreated yarn. Such treated yarn had excellent affinity for acid and direct dyes (e.g. *C.I.* 430, 1076, and 518), whereas untreated yarn had hardly any affinity for them. Particularly brilliant dyeings were obtained with the acid dyes. C. O. C.

Impregnating and Sizing with Latex. A. Dawant. *BP* 677,840

The material (or a plate or plates) is coated with latex containing 60–75% by wt. of dry rubber and is then pressed between the plates to cause the latex to penetrate the material, after which the pressure is suddenly released, thus coagulating the latex within the material. C. O. C.

Cloth with Randomly Arranged Fibres. International Cellucotton Products Co. *BP* 677,545

A bat of fibres is beaten with a spiked roller so as to produce an even dispersion of fibres, which, by means of controlled air currents, is directed against a thermoplastic backing sheet or one to which a liquid adhesive has been applied at an earlier stage. A permanent backing sheet may be dispensed with by directing thermoplastic fibres on to a forming sheet. Subsequent heat or solvent treatment is applied if necessary. J. W. B.

Effects arising from the use of Titanium Dioxide Delustrant in Rayons (VI p. 473).
Raising the Safe Ironing Point of Linear Copolymers containing 1:2:4-Triazole and Peptide Links (VI p. 475).
Stabilisation of Cellulose Triacetate Films against Thermal Oxidative Degradation (XI p. 481).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Preparation of Acidic Oxycelluloses by the Action of Alkaline Hypobromite on Cotton Cellulose. G. F. Davidson. *J. Textile Inst.*, **43**, T 291–T 298 (July 1952).

Samples of cotton linters and Texas cotton sliver, boiled under pressure with NaOH, are treated with alkaline hypobromite, and the change in reagent concentration is used as a measure of oxygen absorbed. All procedures are carried out in the dark to avoid decomposition of hypobromite. Rate of oxidation, loss in weight of the cellulose, and the cuprammonium fluidity, carboxyl content, and copper number of the oxycellulose are measured. There is a progressive loss of weight by the cellulose owing to formation of CO_2 and soluble products. In the early stages of oxidation three atoms of oxygen are required to produce one COOH group, but at an oxygen consumption of 0·25 atom per glucose unit twelve atoms are necessary, owing to the oxidation of soluble products. The method is suitable for making acidic oxycelluloses of COOH content up to 35 millimoles/100 g., but not for high contents such as can be obtained with nitrogen dioxide or periodate–chlorous acid. If the product is to be used for chromatographic columns, it has the advantage of being a powder. J. W. B.

Optical Activity of Cellulose Triacetate. N. M. Bazhenov and M. V. Volkenshteyn. *Izvestiya Akad. Nauk S.S.S.R. Otdel. khim. nauk*, 334–343 (March–April 1952).

Optical activity measurements are made on solutions of penta-acetyl α-D-glucose, octa-acetyl α-cellobiose, and cellulose triacetate in various solvents at various wavelengths and over a range of temp. Determinations are made also of refractive index and dielectric const. By taking the variation of optical activity with temp. into account, it is possible to eliminate the effect of the solvent, if the solute is assumed to be non-associated. In this way a value is obtained for the optical rotatory power of the solute. It should be independent of the temp. if the molecule is rigid, but it does, in fact, vary with temp. in a manner that indicates the presence of a similar type of rotation about bonds in each substance—monomer, dimer, and polymer. It is concluded that in octa-acetyl cellobiose and cellulose triacetate there is rotation of acetyl groups, but no rotation of glucose rings with respect to each other. A. E. S.

Stabilisation of Cellulose Triacetate Films against Thermal Oxidative Degradation. A. A. Freyman, V. A. Bartashev, L. I. Shagalova, V. V. Gannsman, G. P. Marova, and E. L. Ovchinnikova. *J. Appl. Chem. U.S.S.R.*, **25**, 626–633 (June 1952).

On heating in air at 140°C., cellulose triacetate undergoes main-chain breakdown and decarboxylation with liberation of CO and CO_2 at const. rates, which are very greatly reduced by addition to the film of 2% of phenyl-α-naphthylamine or phenyl-β-naphthylamine. A. E. S.

PATENT

Sizes for Paper. N. V. De Bataafsche Petroleum M8. *BP* 678,561

Aqueous emulsions of petroleum naphthenates (i.e. salts of naphthenic acid of acid number < 200 with or without free naphthenic acids of acid number < 200) and aromatic and/or cycloaliphatic mineral-oil extracts are useful sizes for paper and paper pulp. They are much cheaper than rosin sizes and give a more water-resistant effect.

C. O. C.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Aspects of Collagen Structure. J. T. Randall, R. D. B. Fraser, S. Jackson, A. V. W. Martin, and A. C. T. North. *Nature*, **169**, 1029–1033 (21 June 1952).

Work carried out by the authors on the structure of collagen is described briefly, both the interaction of soluble collagen with other molecules, and the polypeptide chain configuration in collagen being discussed. W. J. M.

Bond Forces involved in Chrome Tannage. S. G. Shuttleworth. *J. Amer. Leather Chem. Assocn.*, **47**, 387–403 (June 1952).

The adsorption of eighteen chrome complexes was measured on three types of base-exchange resins (amino, sulphonic acid, and carboxylic acid) and on hide powder. The chrome uptake was then compared with the shrinkage temperature, and it was found that there is a positive correlation with the adsorption on carboxylic-acid-type resin but none with either of the other two. From these results, supplemented by spectrophotometric data, it is concluded that the chief mechanism in chrome tanning is co-ordination of chromium to the carboxy groups of protein side-chains. The ability of protein carboxy groups to displace other ligands from chromium complexes is shown to be inversely related to the dissociation constants of the ligands, thus explaining why chromium formate complexes tan while the acetate complexes do not.

W. J. M.

Isoelectric Points of Chromium Collagen Compounds. K. H. Gustavson. *J. Amer. Leather Chem. Assocn.*, **47**, 425–437 (June 1952).

Various objections to the existing figures for the isoelectric points of collagen have been raised. This has been measured for cationically and anionically chromed collagen and compared with that of the untreated material. Microelectrophoresis, electrokinetic, and dye adsorption techniques have been used, and the results are said to give good agreement. Anionic tannage shifts the isoelectric point to lower pH and cationic tannage to a higher pH. These results strengthen the postulate that collagen is stabilised by the formation of stable cross-links due to complex formation between carboxyl groups of adjacent protein chains. W. J. M.

Practical Experience with Paste Drying. F. Henaker. *Leder*, **2**, 217 (1951): *J. Amer. Leather Chem. Assocn.*, **47**, 446 (June 1952).

Practical aspects of pasting, drying, and washing to remove remaining paste are discussed. Brown leather behaves better than blue, green, or red coloured leather.

C. J. W. H.

Structure of Proteins. J. T. Edsall. *Nature*, **170**, 53–55 (12 July 1952).

The Royal Society discussion (1 May 1952) on "The Structure of Proteins" is summarised, and the most controversial aspects are considered in some detail. These include the α-helix theory of Pauling and Corey; the elastic properties of myosin, epidermin, and fibrin; low-angle X-ray studies on living muscle; and the infrared absorption and dichroism of synthetic polypeptides in the α- and β-forms. J. W. B.

Action of Formaldehyde on Proteins. II—Some Reactions of *N*-Hydroxymethylamides. R. D. Haworth, D. H. Peacock, W. R. Smith, and R. MacGillivray. *J.C.S.*, 2972–2980 (Aug. 1952).

The equilibria between formaldehyde, amides, and *N*-hydroxymethylamides have been studied under alkaline conditions. The *N*-hydroxymethylamides are also found to condense with primary alcohols to form *O*-ethers, with thiols to form sulphides, with carboxylic acids to form esters, and (2 mol.) with primary amines to form tertiary amino derivatives; they do not condense with peptides. The results are applied to a study of the nature of the products formed by the action of formaldehyde upon proteins (cf. *J.C.S.*, 1493 (1950): *J.S.D.C.*, **66**, 606 (1950)).

H. H. H.

Action of Formaldehyde on Proteins. III—Hydrolysis of Formaldehyde-hardened Proteins by Enzymes. R. D. Haworth, R. MacGillivray, and D. H. Peacock. *J.C.S.*, 2981–2987 (Aug. 1952).

The proteins of ground-nuts and soya-beans, and casein, when treated with formaldehyde under acid conditions, are found to be hydrolysed by papain, and the products can be further hydrolysed by trypsin. From the hydrolysate from Ardil protein fibre, six formaldehyde-containing fragments have been isolated by adsorption on charcoal, Zeocarb-215, and cellulose, and analysed by paper chromatography. Acid hydrolysis indicates that aspartic and glutamic acids and at least three other amino acids are associated with these six fragments. The results suggest that *N*-methylenebisamide derivatives of aspartic and glutamic acids play a considerable part, and that lysine and arginine play little, if any, part in the reaction of proteins with formaldehyde under acid conditions.

H. H. H.

Tanning of Fatty Acid and Protein Monolayers by Metal Ions. J. H. Schulman and M. Z. Dogan. *Nature*, **170**, 31–32 (5 July 1952).

Strong interaction between the "di-basic" chromium ion and fatty-acid carboxyl groups is shown by the area of solidification of the molecules and the properties of the fatty acid monolayers on the metal salt solution. The basic chromium ionic unit expands to accommodate the increasing size of fatty acid molecule; this property of the interlinking basic ionic aggregates seems to be greatest with copper and decreases in the order— $Cu^{++} > Cr^{+++} > Al^{+++} > Fe^{+++} > Co^{++}$. Results can be applied to the tanning of protein monolayers over the same optimum pH range; the tanning process works well with Fe, Cr, and Al salts but not with Cu. J. W. B.

Rôle of the Thiol Group in the Formation of Alkaline Protein Gels. H. G. Higgins, D. Fraser, and J. F. Hayes. *Nature*, **169**, 1020–1021 (14 June 1952).

Work has been carried out to measure the sulphide liberated by alkali treatment of casein and also the cysteine and cystine contents of similarly treated casein. As a result, it is apparent that pH 12–13·5 is a critical zone for both the loss in cysteine and the increase in sulphur liberated. From this and other considerations, formation of single sulphur bonds rather than reformation of disulphide linkages seems to be involved in the mechanism of gelation. W. J. M.

PATENTS

Light-stable Phenolic Syntans. American Cyanamid Co. *USP* 2,592,587

The tendency of phenol syntans to darken on keeping and to produce discoloured leathers is much reduced by incorporation of 1–10% of their weight of a vegetable tannin. Leathers tanned by such compositions are readily dyed in clear and light pastel colours. C. O. C.

Finishing Leather. FBy. *BP* 678,614

The products obtained by copolymerising an aqueous emulsion of acrylonitrile and not > 45% of butadiene are useful for finishing leather. Brushing the leather with such an emulsion before applying a collodion coating colour results in excellent adhesion with economy in the coating colour. Added to water coating colours there is such increase in gloss and brilliancy in the finished leather that often there is no need for glazing. C. O. C.

Protecting Furs and Hides from Attack by Insects and Micro-organisms. FBy. *BP* 679,084

The materials are drummed with a cleaning agent, e.g. beech shavings, to which has been added a small amount

of chloromethanesulphontrichloroanilide carried on an inert powder, e.g. beechwood or cork powder of the finest mesh size. The insect- and rot-proof imparted lasts indefinitely. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

PATENTS

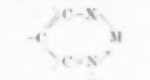
Rubber Antioxidant. American Cyanamid Co. —*BP* 677,584–5

Compounds of formula—

(R^1 = *tert.* Alk of 4–8 C, the tert. C atom being directly bonded to the ring; R^2 = Alk of 1–3 C) are good anti-oxidants which do not increase susceptibility of the rubber to discoloration on exposure to light. C. O. C.

Stabilisers for Synthetic Resins. ICI. *BP* 677,733

Metal chelate compounds containing the group—



(M = metal; X = O or S; R = H, OH, Alk, or Ar; any remaining valences of M are satisfied covalently) stabilise synthetic resins to light and ultraviolet radiation. C. O. C.

Linear Polyamides (VI p. 475).

XIV—ANALYSIS; TESTING; APPARATUS

Standardisation of Methods of Testing Textile Auxiliary Agents—Qualitative Assessment of Oiling Agents with respect to Ease of Removal by Washing. T. Schütte. *Melliand Textilber.*, **33**, 774–777 (Aug. 1952).

Two simple tests are proposed to enable the user of oiling agents to make a rough assessment of the readiness with which the agents will subsequently be removed by washing. In both tests, which are described in detail, a wool fabric is impregnated with a soln. in CCl_4 of the agent and the oil-sol. dye Sudan Violet BR. After drying, the fabric is treated with cold Na_2CO_3 soln.—in one method by shaking with the soln., and in the other by allowing a given vol. of the soln. to syphon through a hanging strip of the fabric. The amount of residual oil is assessed from the colour of the fabric, and it is shown, by a solvent extraction method, that a reliable estimate is obtained. A. E. S.

One-dimensional Paper-chromatographic Technique for Separation of Phenols, Phenolic Acids, and their Derivatives. J. A. Durant. *Nature*, **169**, 1062–1063 (21 June 1952).

A technique for separating the above compounds has been worked out. The chromatograph is carried out on paper impregnated with a buffer, and characteristic colours are developed by spraying with Millon's reagent, drying, and respraying. Development takes ~ 5 hr. at 35°c. A table of 13 substances, including the cresols, gives their R_F factors and the colours developed with Millon's reagent. W. J. M.

Colorimetric Method of Estimating "Diazols". V. E. Rostovtsev and V. V. Gromova. *Tekstil. prom.*, **12**, 32–33 (May 1952).

2-5-Aminonaphthol-7-sulphonic acid (J acid) is recommended as coupling reagent for the colorimetric estimation of diazonium salt in a soln. of a commercial azoic component. A. E. S.

Shutter-illumination Device for Studying Phototropic Dyes. J. H. Gould and W. R. Brode. *J. Optical Soc. Amer.*, **42**, 380–382 (1952); *Chem. Abs.*, **46**, 7440 (25 Aug. 1952).

To study the phototropism shown by some monoazo dyes, a shutter and a sample illuminator were made for use with a Cary recording quartz spectrophotometer. This enables the dye soln. to be exposed to a strong light source during the dark periods of the chopping shutter of the instrument, and permits study of solutions which show very rapid phototropic change. C. O. C.

Ionophoretic Separation of Porphyrin Pigments. S. C. Papastamatis and J. E. Kench. *Nature*, **170**, 33–34 (5 July 1952).

Evaluation of Aluminium Pigments. A. Knoll. *Official Digest Federation Paint & Varnish Production Clubs*, (318), 447–451 (1951); *Chem. Abs.*, **46**, 7787 (25 Aug. 1952).

The layer of "grease" responsible for the leafing properties of Al pigment remained after extraction with hot acetone, and could be removed only by extraction with xylene at 140°c. or by dissolving the metal with HCl. This tightly held layer on analysis appeared to be Al distearate, which is in agreement with Trillat's X-ray diffraction study. The coarser leaf has higher reflective power than the finer one; the 60° glossmeter used could not show the smoother texture of the fine-pigment film; surface tension of the vehicle did not affect the reflectance factor; and higher pigment : vehicle ratios increased total reflection. C. O. C.

Analysis of Pigments in Coatings. I—White Pigments. II—Red Pigments. H. Kittel and W. Reimold. *Deut. Farben-Z.*, **5**, 424–425 (1951); **6**, 127–131 (1952); *Chem. Abs.*, **46**, 6847 (25 July 1952).

I—Treatment with dil. and conc. H_2SO_4, HCl, and HNO_3 and 20% NaOH distinguishes $BaSO_4$ (sol. only in conc. H_2SO_4) from ZnS (sol. except in NaOH), ZnO (sol. in all reagents), white lead (sol. except in dil. H_2SO_4), TiO_2 (sol. only in hot conc. H_2SO_4 and partly in conc. HCl), and $PbSO_4$ (sol. in conc. H_2SO_4, partly in other conc. acids, and sol. in NaOH). TiO_2 and $BaSO_4$ are dissolved by hot conc. H_2SO_4, but only the former stays in solution after dilution with water. TiO_2 is separated from Zn also in such a solution by precipitation with NH_4OH and calcination of the ppt. to TiO_2. $PbSO_4$ and $BaSO_4$ are separated by NaOH, which dissolves only the former.

II—Red ferric oxide (I), cadmium red (II), molybdate red (III), Pb_3O_4 (IV), and chrome orange (V) are distinguishable by their solubility in acids and 20% NaOH, as follows—In conc. H_2SO_4 all except I are sol. hot, but I is sol. on dilution. In dil. H_2SO_4 (1 : 1) I is sol. hot, II and IV are insol., and III and V are sol. hot but ppt. $PbSO_4$. In conc. HCl II, III, and IV are sol. cold, I is sol. hot, and V is reduced. In dil. HCl (1 : 1) all are sol. hot but V is reduced. In conc. HNO_3 III, IV, and V are sol. cold, I is sol. hot, and II is sol. cold with precipitation of S and Se. In dil. HNO_3 (1 : 1) I is insol., II and V are sol. cold, II is sol. hot with precipitation of S and Se, and IV is partly sol. hot with precipitation of PbO_2. In 20% NaOH III and V dissolve cold, IV dissolves hot, and I and II are insol. Analysis of mixed pigments is first carried out to detect the red, white, and other pigments probably present. The determinations are then carried out in a manner most suited to the combinations present. Such schemes are suggested for combinations of I, II, III, IV, or V with barytes, lithopone, and ZnO–zinc white. Presence of TiO_2, cadmium red, or HgS is considered rare and unlikely because of price. Organic pigments can be estimated by ashing. Zn pigments ashed in presence of Co drier turn green. C. O. C.

Estimation of Free Amino Acids using a Microdiffusion Technique. W. B. McConnell. *Canadian J. Chem.*, **30**, 522–528 (July 1952).

The reaction between ninhydrin (triketoindane hydrate) and α-amino acids stoichiometrically liberates CO_2, which is absorbed by alkali placed in the inner chamber of a diffusion cell. $BaCl_2$ is added to precipitate the carbonate, and excess alkali titrated with HCl. The method has been used to estimate glycine, leucine, alanine, glutamic acid, valine, arginine, tryptophan, aspartic acid, and cystine, also to follow the enzymatic release of amino acids from proteins. In the example given gelatin is incubated with trypsin, and the free amino acids are estimated. A. H.

Estimation of the Whiteness of Optically Whitened Textiles. E. C. Caspar. *Melliand Textilber.*, **33**, 518–521 (June 1952).

Bleached cotton and wool textiles are treated with various fluorescent whitening agents, or with ultramarine, and their reflection spectra relative to an $MgCO_3$

surface are determined with the aid of a Sigrist reflection spectrophotometer. In this instrument, a single photocell is used, but effects due to variations in the light source and cell characteristics are virtually eliminated. The pattern and standard surface are diffusely illuminated in an Ulbricht sphere having a matt aluminium surface (high ultraviolet reflectivity), and light from the surfaces under comparison passes out and through an interference filter before reaching the photocell. The cell receives alternately light from the pattern and light from the standard surface, and when the intensities of the beams are unequal, there is a "flicker" effect in the current generated by the cell. An electronic device enables the flicker to control the current to an electric motor, which operates a variable diaphragm, placed in the light path from the standard surface, until the flicker effect is eliminated, i.e. until the light beams become equally intense. The relative reflectivities of the surfaces can then be read off on a drum that is coupled to the diaphragm. The specification of the source of light is of great importance, if results are to be comparable with visual results using daylight. The lack of a suitable source is at present an obstacle to the development of the method for precise quantitative purposes; the most satisfactory source found is the blended radiation from a Philips ciné projector lamp and a Philips Philora ultraviolet lamp, and it is shown that results obtained using this source can be closely correlated with visual observations. For comparison, determinations using only visual light are made. Photospectrographic curves and curves showing the variation of the reflection intensity at various wavelengths with the concn. of agent are given for agents of varying effective hue, and the curves are discussed in their relation to visual estimations of the whitening effect and of the optimum concn. of agent.

A. E. S.

Practical Method for Measuring and Expressing Colour. J. Christensen. *Norsk Skogindustri*, **5**, 265–270 (1951): *Chem. Abs.*, **46**, 6384 (10 July 1952).

The colour of paper is measured with a Photovolt reflection meter, using blue, green, and amber filters. The readings are plotted on a 3-axis system, from which, by simple geometric procedure, values for tone, strength, and brightness are computed. These three characteristics define each and all coloured surfaces. Examples (data in graphs and tables) show how the method can be used to define colour of samples, to match a given colour, and to evaluate dyes.

C. O. C.

Measurement of Reflectance of White Sugar. T. R. Gillett and P. F. Meads. *Anal. Chem.*, **24**, 829–832 (May 1952).

A photoelectric reflectometer has been developed to measure the apparent colour of dry white sugars; it employs a balanced photoelectric circuit and the principle of diffuse reflectance. Light from a single lamp source impinges at 45° on two apertures, directly below which (and on the same side as the light source) are the photocells screened by colour filters. Normally reflected light does not reach the cells, but only that due to diffuse reflection. The instrument is first balanced by covering the apertures with reference standards (white glazed tiles) and adjusting a balancing rheostat to give a null galvanometer reading. One tile is then replaced by the sugar sample on a glass plate (which does not in itself affect the result), and a reading is obtained by adjusting a slide wire to restore balance. The instrument is described fully, and the effects of such factors as grain size, lustre, and colour distribution on the reflectance are discussed.

J. W. D.

Determination of the Fat Content of Silk-soaking Emulsions. F. Bryant. *Textile Research J.*, **22**, 433–434 (June 1952).

A new method is described for determining the fat content of the aqueous emulsions of a sulphated oil used for soaking raw silk skeins. A Babcock test bottle is used as before, but the emulsion is broken using a hot alcohol solution containing calcium chloride and water.

A. B.

Luminol as a Chemiluminescent Indicator in Photometric Titration of Solutions of High Opacity. F. Kenny and B. B. Kurtz. *Anal. Chem.*, **24**, 1218–1219 (July 1952).

A photometer is used to locate the chemiluminescent end-point of an acid–alkali titration which employs luminol as indicator, and is carried out in a dark-chamber titrimeter. Hydrogen peroxide is used to oxidise the luminol, and haemoglobin is added to catalyse the oxidation; these indicator constituents are added to the alkaline soln., whilst the luminol itself is dissolved in the acid, thus obviating the danger of a false end-point resulting from the depletion of one of the indicator constituents. The method, which is fully described, gave results of satisfactory precision when tested with soln. containing sufficient Indian ink to render them opaque to a 200-watt lamp.

J. W. D.

Improved Colour-difference Formula, with Applications to the Perceptibility and Acceptability of Fadings. I. H. Godlove. *J. Opt. Soc. Amer.*, **41**, (11), 760–772 (1951): *J. Textile Inst.*, **43**, A 416 (June 1952).

A simple formula for computing fadings or other colour differences from Munsell notations has been derived from one simple assumption. Experimental data for the relative visual magnitudes of the Munsell "value" and chroma steps have been incorporated into the formula. It has been tested with several sets of experimental data, and found valid even for some large differences. A table of constants facilitates quick application. As previously reported (see *J. Textile Inst.*, **43**, A 311 (1952)), the formula has been applied to the computation of the colour differences of a great many dyes changing in depth only ("on-tone" fading).

C. J. W. H.

Instruments for Measuring Crease Resistance. A.A.T.C.C. *Amer. Dyestuff Rep.*, **41**, P 419–P 423 (7 July 1952).

Data are presented of tests for crease resistance carried out on different fabrics and in different laboratories, using a roller pressure tester (American Cyanamid Co.) and a wrinkle recovery tester (Monsanto). Both instruments are accepted as tentative standards.

J. W. B.

Testing Water Repellency. E. M. Kärrholm. *J. Textile Inst.*, **43**, P 263–P 289 (June 1952).

The Swedish rain tester is described, and some test methods for water repellency are compared. The new tester is of the artificial rainfall type, the drops being formed by glass capillaries. Both absorption and penetration of water are determined for each sample. The influence of various factors, e.g. temperature, hardness, pH, rigidity of support, and height of fall are discussed. The test gives a higher sensitivity for wool fabrics than the dynamic absorption test, in which fabrics move in water. It is suggested that low drop intensity is preferable for fabrics with a compressible or raised surface.

J. W. B.

Volumetric Determination of Melamine as Oxalate. J. R. Steele, J. H. Glover, and H. W. Hodgson. *J. Appl. Chem.*, **2**, 296–298 (June 1952).

The solution of melamine (0·1 g./20 ml.) is added with stirring to 5 ml. of a freshly prepared 20% solution of oxalic acid in ethanol, and the mixture is cooled to < 4°C. for 30 min. The precipitated melamine mono-oxalate is filtered and washed 5 times with 5 ml. of precooled ethanol, dried, dissolved in dil. H_2SO_4, and titrated at 70°C. with 0·5 N-$KMnO_4$ (1 ml. ≡ 0·00315 g. melamine). The method is accurate to 0·3% melamine, and small quantities of cyanogen compounds related to melamine have only slight effect.

W. K. R.

Identification of Polyacrylic Compounds in the Analysis of Finishes, Sizes, and Fibres. H. Rath, G. Nawrath, and E. Schönpflug. *Melliand Textilber.*, **33**, 636–639 (July 1952).

The compositions of 15 commercial polyacrylic and polymethacrylic preparations used in sizing and finishing are listed. A scheme of analysis is described for the identification of such compounds on the fibre. In the preliminary hydroxylamine test for an ester group (formation of ferric complex of a hydroxamic acid) polyacrylic and polymethacrylic esters are distinguished by their colorations (brownish orange and reddish violet respectively). Polyacrylic esters, unlike polymethacrylic esters, are readily hydrolysed by alcoholic KOH and can be estimated quantitatively in the hydrolysate as barium polyacrylate. If a polyvinyl ester is present, polyvinyl alcohol is formed and can be detected by its iodine reaction. Polymethacrylic esters are best detected in the residue from a suitable saponification treatment; this residue is quantitatively depolymerised by heat, and the nature of the monomer

may be confirmed by a number of reactions. Polyacrylic esters are not quantitatively depolymerised by heat, but enough monomer is formed to allow its detection by reaction with phenylhydrazine, which leads to a dark green coloration. Polyacrylonitrile is identified by treatment with sodium thiosulphate, which gives thiocyanate, detected as the red ferric complex. The identification also of the alcohol components of polyacrylic and polymethacrylic esters is considered. A. E. S.

Determination of the Resistance of Wool and other Keratinous Materials to Attack by Moths or Beetles after Dry Cleaning or Washing. Schweizerische Normen-Vereinigung, *SNV* 95 9 11; *Textil-Rund.*, **7**, 278 (June 1952).

Knitted fabric, felt, or cloth (10 × 20 cm.), yarn (10 g.), or loose material (5 g.) is dry-cleaned in trichloroethylene, benzene, or perchloroethylene for 30 min. at 20 ± 2°c. in a 40 : 1 liquor with agitation or squeezing and air-dried at a max. temp. of 80°c.; or washed in 5 g. alkali-free soap per litre for 30 min. at 40 ± 2°c. in a 50 : 1 liquor by hand or using a mechanical device as described in SNV 95 8 21, rinsed, hydroextracted, and dried in warm air at a max. temp. of 60°c. The dry-cleaned or washed material is then tested for resistance to attack by moths or beetles according to the tests given in SNV 95 9 01, 95 9 02, and 95 9 10. B. K.

Application of Chromatography to the Paper Industry. A. F. Williams. *Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*, **33**, 85–106 (Feb. 1952).

Qualitative and quantitative aspects of methods for the separation of small amounts of metal ions on paper strips or columns of cellulose powder using organic solvents are described. The application to the analysis of paper ash, the use of chromatography in organic separations, and its possible use in separating and identifying dyes are also considered. S. V. S.

Determination of Tricresyl Phosphate in Cellulose-finished Leathers. L. M. Hill. *J. Soc. Leather Trades Chem.*, **36**, 113–119 (April 1952).

The standard volumetric phosphomolybdate method for tricresyl phosphate gives inconsistent results when applied to treated vegetable-tanned upholstery leather, but the colorimetric molybdivanadophosphoric acid method is simple, accurate, and reproducible. The leather may be digested with sulphuric and nitric acids in presence of potassium nitrate, or a mixture of sulphuric, nitric, and perchloric acids, either alone or in presence of a sodium molybdate catalyst. The colorimetric technique is as described by Simmons and Robertson (*Anal. Chem.*, **22**, 294 and 1177 (1950)). A. S. F.

Determination of the *iso*Cyanate Group in Rubber-bonding Agents. A. G. Williamson. *Analyst*, **77**, 372–374 (July 1952).

Compounds with more than one *iso*cyanate group per molecule are important in promoting good adhesive bonds between rubber and textiles. The *iso*cyanate is treated with di-*n*-butylamine in a solvent comprising monochlorobenzene (30 vol.), methanol (100 vol.), and water (13 vol.), and the excess amine is titrated electrometrically with HCl in the same solvent, using the glass electrode. Results obtained with preparations such as Vulcabond TX and Desmodur T showed an estimated standard deviation of only 0·5%, as compared with one of 1% associated with a similar colorimetric procedure. The substitution of piperidine for di-*n*-butylamine proved unsatisfactory. The method is given in full. J. W. D.

Determination of Fluorine in Polytetrafluoroethylene. J. Haslam and S. M. A. Whettem. *J. Appl. Chem.*, **2**, 339–344 (June 1952).

The ground sample is fused with sodium peroxide in a Parr bomb, and the aqueous extract of the melt is distilled with perchloric acid in the presence of silica. The fluoride in the distillate is titrated at a controlled pH of 3·0 with thorium nitrate solution using sodium alizarinsulphonate as indicator. The end-point is determined by comparison with a colour standard containing thorium fluoride, cobalt sulphate, and potassium dichromate. The thorium nitrate is standardised against sodium fluoride which has been submitted to the above procedure. W. K. R.

PATENTS

Continuous Indication of Denier by Conductivity Method. British Nylon Spinners. *BP* 674,905

Continuous indication of the cross-sectional area of a running filamentary structure is provided by a cell containing an electrolyte, e.g. aq. NaCl, and electrodes in an A.C. circuit adapted to measure the resistance between them. The cell is divided between the electrodes by a septum with a perforation through which the thread is passed after it has been wetted. Wetting may be done either in a separate shallow U-tube containing solution identical to that in the cell, or by having a more subdivided cell in which the thread passes through two preliminary perforated septa, the resistance being measured across the third. A stable oscillator provides A.C. through a Wheatstone bridge; incorporation of a phase-discriminating detector permits direct indication of denier. J. W. B.

Measurement of Temperature Distribution on a Solid Surface. Eastman Kodak Co. *BP* 675,043

The surface is coated with a thin layer of a phosphor whose brightness and/or colour varies according to its temperature, the changes in brightness being recorded either photographically or by use of a photoelectric cell. C. O. C.

Iron-containing Oleines (III p. 463).
Structure of Hydroxyazo Dyes and their Tendency to Change Hue on a Magnesium Hydroxide Surface (IV p. 465).
Influence of the Adhesion of Printing Inks on their Properties (V p. 471).
Swelling and Swelling Anisotropy of Rayon (VI p. 472).
Recent Investigations in Wool Chemistry (VI p. 474).
Lytax Textiloscope (IX p. 478).
Raising of Textile Fabrics (X p. 479).

XV—MISCELLANEOUS

Colouring of Metals—Historical Aspects. W. W. G. Hübner. *Ciba Review*, **8**, (92), 3298–3301, 3328 (June 1952).

A historical survey is followed by a brief account of the methods now in use—viz. (1) *Mechanical colouring*, i.e. the hammering of coloured materials into soft or engraved metal, enamelling, varnishing, or bronzing. (2) *Chemical colouring*, i.e. transformation of the surface by chemical action. (3) *Electrochemical colouring*, in which the metal is treated in suitable liquids by an electric current. (4) *Electroplating*. 11 References. C. O. C.

Surface Treatment and Colouring of Aluminium. W. W. G. Hübner. *Ciba Review*, **8**, (92), 3313–3328 (June 1952).

The various chemical and electrical processes for treating aluminium surfaces are described, details of the application and results obtained by the processes now in use for anodic surface treatment being tabulated. The colouring of oxidised aluminium surfaces with inorganic and organic colouring materials from aqueous and organic-solvent solutions is described. C. O. C.

The Macintoshes and the Origins of the Chemical Industry. D. W. F. Hardie. *Chem. and Ind.*, 606–613 (28 June 1952).

An account of the part played by the Macintosh family and particularly Charles Macintosh in the chemical revolution that formed part of the industrial revolution that occurred in Great Britain in the latter half of the 18th and the first half of the 19th centuries. The parts they played in the development of the cudbear, alum, Prussian blue, bleaching, waterproofing, calico printing, and Turkey Red dyeing industries in Scotland are dealt with, and evidence is provided suggesting that Charles Macintosh and not Charles Tennant was the real inventor of bleaching powder. C. O. C.

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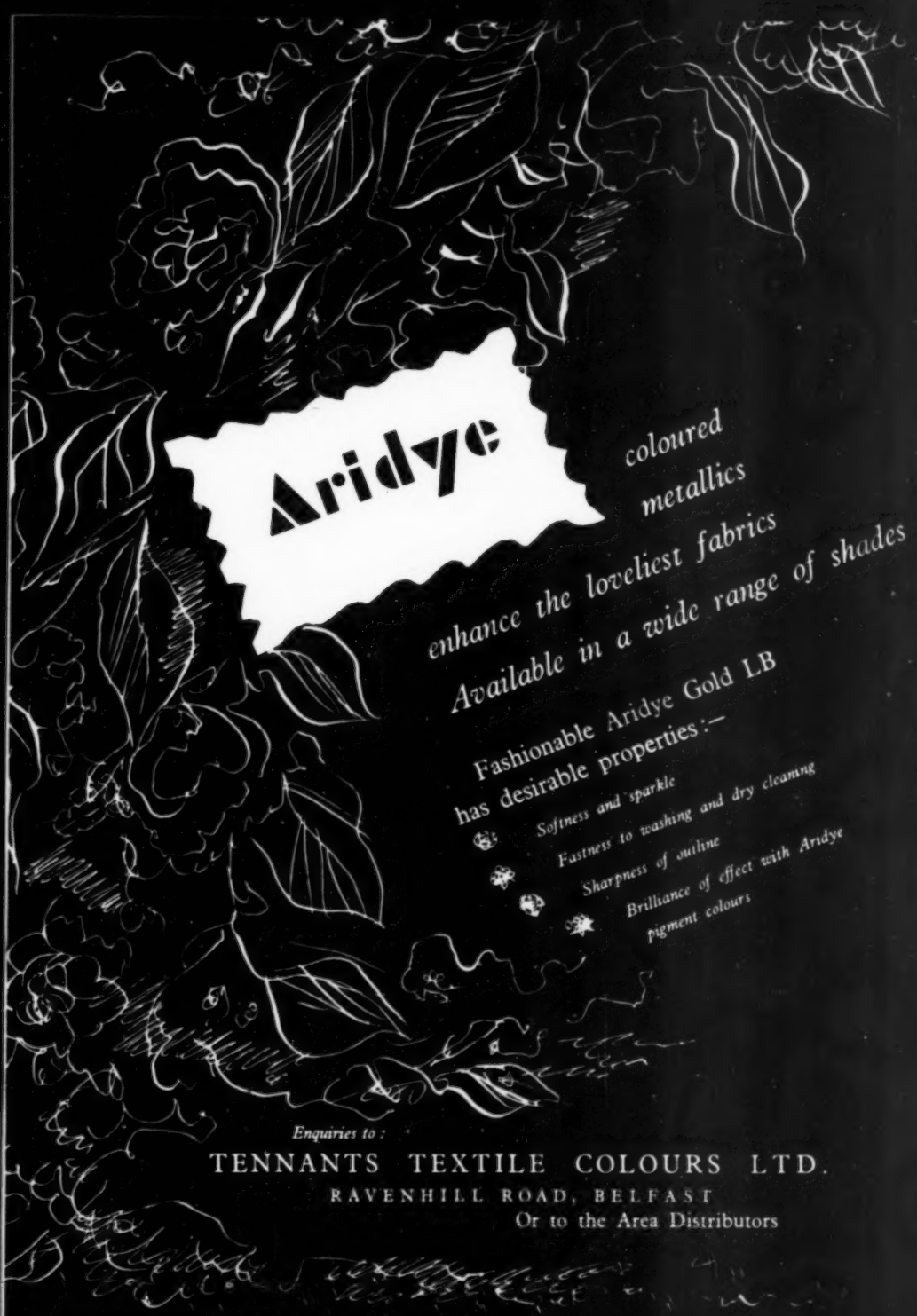
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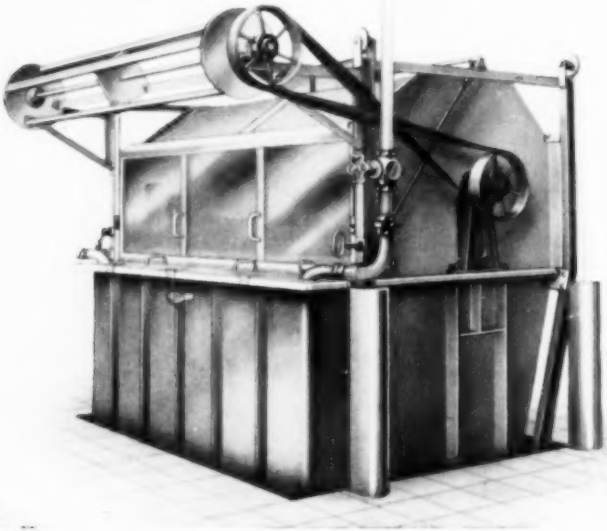

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FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page* xii

Manchester Junior Branch—*continued*

1953

Monday 2nd Feb. — *Dyeing and Finishing. The Public's Reaction to Fastness Properties* J. S. Ingham Esq.

Monday 2nd March — Title to be given later.

LEEDS JUNIOR BRANCH

All meetings to be held in the Colour Chemistry Lecture Theatre, The University, Leeds, 2 on Tuesdays at 4.0 p.m.

1952

25th Nov. — C. O. Clark, Esq., F.T.I. *Historical Records*

1953

20th Jan. — J. Boulton, Esq., M.Sc.(Tech), F.R.I.C., F.T.I. (Courtaulds Ltd.). Title to be announced

17th Feb. — G. G. Taylor, Esq., B.Sc., A.Inst.P. and J. C. Brown, Esq. (The Clayton Aniline Co. Ltd.). *The Uses of Microscopy in Textile Dyeing and Finishing*

3rd March — D. Hanson, Esq., B.Sc., F.R.I.C. (J. Crowther & Sons Ltd.). *A Chemist in the Woollen Industry*

WEST RIDING SECTION

All meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1952

Tuesday Nov. 25th — Dr. R. L. Wormell (Courtaulds Ltd.). *Casein and Peanut Protein Fibres* (Joint lecture with the Yorkshire Section of the Textile Institute). Midland Hotel, Bradford

Thursday 27th Nov. — H. R. Hadfield, Esq. (Imperial Chemical Industries Ltd.). *A Review of the Dyeing of Terylene Polyester Fibre*

Thursday 11th Dec. — FILM EVENING. Ladies to be invited. (Joint meeting with Yorkshire Section Textile Institute)

1953

Friday 16th Jan. — LADIES' EVENING

Thursday 29th Jan. — Lecture. Details later

West Riding Section—*continued*

1953

Thursday 12th Feb. — D. P. Raper Esq. (Imperial Chemical Industries Ltd). *Wool Dyeing Faults and their Correction*

Monday 23rd Feb. — N. R. Hjort, Esq. *Water Treatment.* (Joint lecture with the Halifax Textile Society). Alexandra Cafe, Halifax

Thursday 26th Feb. — J. V. Summersgill Esq. (Geigy Co. Ltd.). Title later

Tuesday 3rd March — Dr. H. Baines (Kodak Ltd.). *Colour Photography.* The University, Leeds

Thursday 12th March — Dr. J. F. Gaunt (Patons & Baldwins Ltd.) *A Study of the Afterchrome Process of Dyeing Wool*

Thursday 26th March — ANNUAL GENERAL MEETING

BRADFORD JUNIOR BRANCH

All meetings held in the Bradford Technical College at 7.15 p.m.

1952

Thursday 27th Nov. — Visit to Salts (Saltaire) Ltd.

Tuesday 9th Dec. — FILM EVENING

1953

Monday 19th Jan. — J. W. Fisher, Esq., B.Sc., F.R.I.C. (British Celanese Ltd.). *Synthetic Fibres*

Wednesday 4th Feb. — Visit to Messrs. Montague Burton Ltd., Hudson Road Mills, Leeds

Wednesday 18th Feb. — T. Green, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of pH and its Importance in Relation to Textile Processing*

Thursday 26th Feb. — JUNIOR BRANCH DANCE at the Queens Hall

Friday 13th March — STUDENTS' ANNUAL COMPETITION EVENING

Thursday March 26th — FILM EVENING

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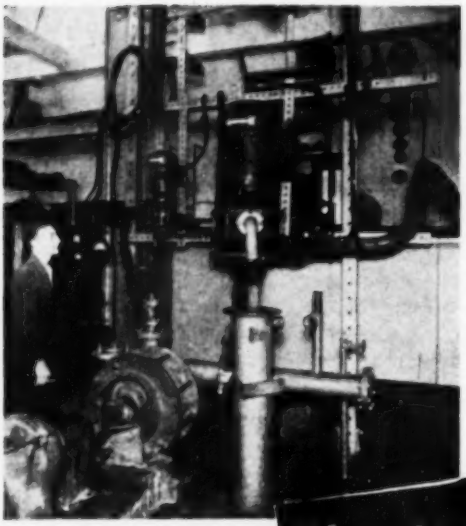

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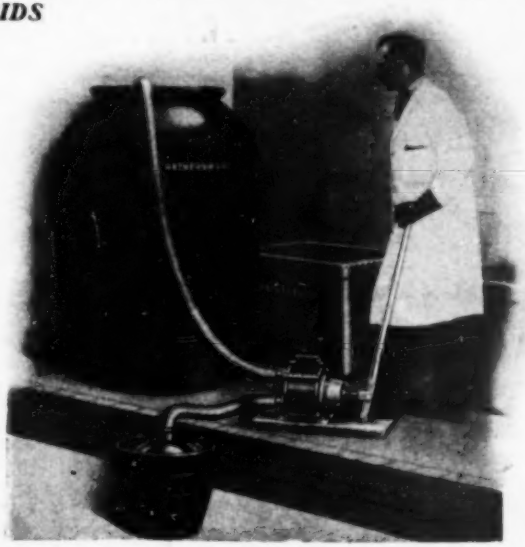

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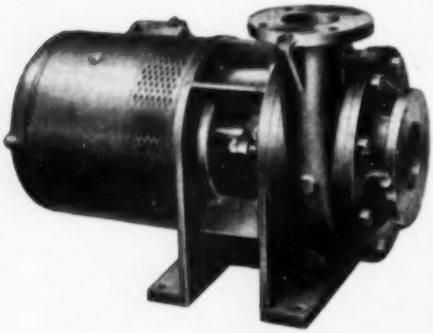

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Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

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